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METHODS OF RADIOLOGICAL TEST FOR WATER

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UDC 628·1/·3 : 543·3 : 543·53

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Indian Standard

METHODS OF RADIOLOGICAL TEST FOR WATER

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(*Continued on page* 2)

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(*Continued on page* 51)

Indian Standard

METHODS OF RADIOLOGICAL TEST FOR WATER

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 31 October 1985, after the draft finalized by the Water Sectional Committee had been approved by the Chemical Division Council.

0.2 Radioactive materials are found in the environment originating from a number of sources — natural and man made. In nature they are produced by cosmic rays and may find their way into water courses with rain fall and run off (radioisotopes of lighter nuclides like tritium, beryllium and carbon). Naturally occurring radioactive elements such as uranium and thorium along with their daughter products, are also present in the soil, earth crust and rocks, which may also enter into the water bodies through weathering. The man made radionuclides, however, enter the environment, mainly from nuclear test fall outs, release of radioactive waste from nuclear power plants, nuclear facilities and other industries like pharmaceuticals and agriculture, that employ radioactive isotopes.

0.3 It is worthwhile to note here that in addition to the various sources described above, a few radioactive nuclides like potassium-40, radium-226, and 228, lead-210 and carbon 14 are inherent in the human body itself.

0.4 One of the various pathways for the radionuclides to enter human body is ingession through drinking water and food materials raised on contaminated water. The radionuclides are absorbed in the body and follow similar metabolic course as their stable isotopes. For example tritium taken in as oxide in the form of tritiated water mixed with the body fluid is rapidly distributed over the whole body. Such materials give rise to whole body dose similar to that received from external radiation except that they continue irradiation till they are substantially decayed or eliminated from the body by biological processes. Some nuclides are, however, selective such as alkaline earths (calcium, strontium and barium) which go to the skeleton. These are mostly *beta*-emitters thus giving rise to radiation damage that is quite localised. The radioactive isotopes of heavy metals like radium-226 and 228, and plutanium-239, 240 and 242 are also bone seekers. Another highly selective radionuclide is iodine-131 which quickly gets concentrated in the thyroid gland weighing only a few grams.

0.5 Some radioactive materials which are insoluble or get slowly dissolved in the body fluid, irradiate lungs and gastrointestinal tract until they are decayed or eliminated from the body.

0.6 The harmful effects of radionuclides which enter into the human system are considered to be due to ionization of the atoms in the molecules of the chemical constituents of the cells which are basic building units of body tissues. As the result of ionization some of the molecules of the cell constituents get damaged or disintegrated and hence cannot function normally. Also, the products of molecular disintegrations tend to clog and poison the cell. If only relatively few atoms in a cell are ionized it may recover from the damage without much difficulty. However, if a comparatively larger number of ionizations occur the cell may become unable to carry on its activities and will die. The nuclear radiation may cause (a) swelling of the nucleus and sometimes of the entire cell, (b) increase in the viscosity of the cell fluid, (c) increase in the permeability of the cell membrane, (d) delay or prevention of cell division and (e) injury to the chromosomes which are microscopic thread like bodies that carry the genes of heredity.

0.7 Radionuclides of interest in water and waste water have been identified on the basis of those present in the natural environment as well as those resulting from human activities. They are basically α-emitters (along with their daughters) and β-emitters. The naturally occurring α-emitters of interest include radium-226, polonium-210, radon-220 and 222 and isotopes of uranium and thorium. The β-emitting nuclides include radium-228, lead-210 and carbon-14. On the other hand the man made radionuclides of interest include fission products and neutron activation products from nuclear reactors. These are tritium, cobalt-58 and 60, strontium-89 and 90, iodine-129 and 131, cesium-134 and 137, plutonium-239 and 240 and americium-241.

0.8 In order to keep a proper inventory of the various radionuclides entering into the water bodies and in turn into the human system, it is essential to adopt rigorous sampling and analytical procedures. For the general guidance of the water supplier, public health engineer or analyst and the competent authority, the International Commission on Radiological Protection (ICRP) and World Health Organisation (WHO) Working Group have recommended maximum permissible concentrations of various radionuclides that may be present in the water bodies.

0.9 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

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*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes methods of sampling of radionuclides in water, methodology of radiochemical separations of individual nuclides, instrumentation for *alpha* and *beta*-counting and data processing. This standard is applicable to all types of water.

2. SAMPLING

2.0 The collection of valid samples is equal in importance to accurate analysis in carrying out a programme for evaluating contamination of man and his environment. Unlike step by step procedures for chemical and radiochemical analysis, no such steps can be described for general process of sampling. The samples should be representative and the analysis should be accurate, otherwise the entire process becomes invalid.

2.0.1 The concentration of a particular radionuclide, for example, in the water bodies, may change with time and location. The crucial decisions in planning a sampling operation are usually (a) number of sites to be sampled, and (b) frequency of sampling. Water samples taken for radiochemical analysis may either be analysed directly as individual samples or they may be combined into composites representing a group of locations and a period of time or both. This compositing is usually carried out to reduce the load on laboratory while maintaining the representative nature of the sample.

2.1 In taking the waste water samples it should be kept in view that the concentration of radionuclides is highest as it leaves the source and hence the detection of any contamination should be made at the source, that is, at the discharge point itself.

2.1.1 The storage of water samples prior to analysis requires some careful thinking so as to avoid loss of certain radionuclides. Adsorption of most of the radionuclides is generally less on polyethylene than on glass surfaces. Some exceptions have, however, been reported but all of these may be eliminated by addition of an acid, a carrier or a complexing agent to the sample.

2.1.2 The general subject of water sampling is usually considered as a unit even though sampling of a river or of a tap water are certainly widely different in techniques. It is assumed, however, that water is a homogeneous medium and that sampling is thereby quite simplified.

2.1.3 Usually the samples for evaluating water radioactivity have to be quite large so as to obtain sufficient quantity for analysis. In addition, continuous sampling has many advantages over a grab sample. Thus in the sampling of process streams, waste streams rivers and stagnant waters, it is generally desirable to have a pump sampler that, although collecting at a slow rate, is able to provide a sufficient amount of sample over a desired period. Where continuous sampling is not practical (field sampling), it is still possible to take 'grab' samples at reasonable intervals of time and composite them into a final sample for measurements. In case of a stream, for example, the composite sample may consist of either several samples taken at a time at various locations across the profile, or several sub samples collected at one location over a period of time.

2.2 Drinking Water

2.2.1 All sampling equipment coming into contact with the sample should be rinsed with the water to be sampled.

2.2.2 The water at the point of sampling should be agitated before taking the sample in case of well waters and lakes. In case of tap water, water should be allowed to run for a few minutes before a sample is collected.

2.2.3 Sample should be collected about a foot below the surface of a stream, well, or a lake.

2.2.4 Provided radioactive effluents are not discharged into water bodies, radionuclide levels are usually very low in drinking water supplies. Hence sufficiently large amounts of samples, of the order of 20 to 30 litres are required, to be collected.

2.3 Waste Water

2.3.1 The variation in the composition and concentration of radionuclides being quite frequent in the effluents, 'grab' samples are generally not recommended. Apart from the variations in the quality of the effluent, the quantity being discharged also varies considerably with time. A proportionate sample is therefore a combination of aliquots drawn continuously from the effluent stream. The sampling units which draw aliquots of the effluent at regular intervals are now commercially available. However, the main drawback of such samplers is that they require electric power to run which may not be available for the field sampling. Also fluctuations in the quantity of the effluent do not get reflected in the aliquots as they are drawn at regular intervals irrespective of the flow rate.

2.3.2 A sampler has been developed for continuous sampling of effluent streams. The sampler consists of a paddle wheel of about 120 cm diameter installed in the stream to be sampled. The wheel size depends upon the drain dimensions and other site conditions. The wheel is rotated about a horizontal axis by the stream itself without any external power. The movement has to be as frictionless as possible, and is achieved by the use of ball bearings. A tiny vial about 2-ml capacity attached to the rim and properly counterbalanced serves to collect the aliquotes of the effluent flowing through the stream. The wheel is kept in motion by the paddles. The rate of rotation of the wheel depends on the flow of the stream. Thus the sample collected will be proportional to the quantity of effluent discharged, that is, flow rate and time. The continuous samples thus collected may be further composited to reduce the volumes.

2.3.2.1 The sampler described above, is however, not suitable for sampling of pipe discharges.

3. INSTRUMENTATION

3.1. Alpha Counter

3.1.1 The *alpha* particles are counted either by windowless proportional counter or a silver activated zinc sulphide coated scintillation counter. The scintillator is a thin layer of powdered zinc sulphide crystals doped with silver coated directly on photomultiplier tube or on lucite which serves as light pipe. This is covered by an extremely thin light tight, both side aluminized, cellulose acetate film (1·5 mg/cm^2). When the *alpha* particles strike the zinc sulphide (ZnS) silver (Ag) crystals a light flash is produced, which in turn is seen by a photomultiplier tube, and then amplified, processed and counted by different units. A schematic diagram of the *alpha* counting equipment is given below:

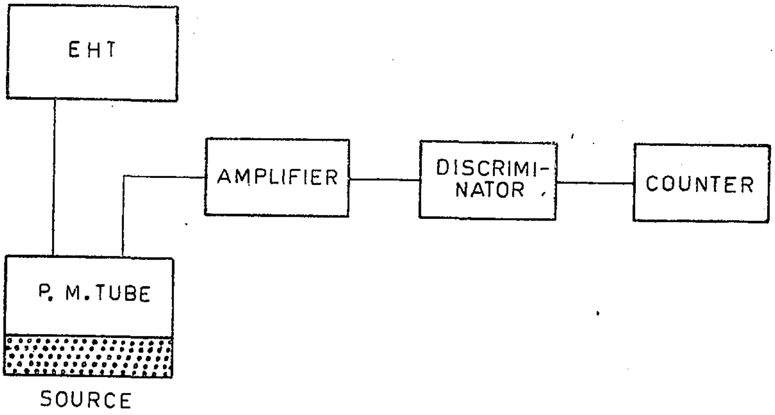

FIG. 1 SCHEMATIC DIAGRAM OF α-COUNTING EQUIPMENT

3.1.2 The efficiency of a good *alpha* counter should approach 50 per-cent. Lesser efficiencies are due to self absorption within the source itself and the greatest problem is source preparation so as to produce an essentially weightless source to minimize self absorption. Generally the carrier should be limited to less than 1 mg/cm^2. For this reason electroplating of *alpha* emitters from carrier-free or low carrier solution is preferred. Fortunately uranium, polonium, lead and many other *alpha*-emitters can be readily electroplated.

3.1.3 *Gamma*-rays are seldom absorbed in the zinc sulphide (ZnS) silver (Ag) layer because it is very thin. *Beta* particles are partially absorbed producing very small pulses while *alpha*-particles are completely absorbed producing very large pulses. The discriminator is set so that it accepts the large voltage pulses produced by the *alpha*-particles only. The operational details are provided with the commercially available instruments.

3.1.4 For detailed study of the *alpha* emitting radionuclides, surface barrier (solid state) α-spectrometer is used. A schematic diagram of the solid state α-spectrometer is given below:

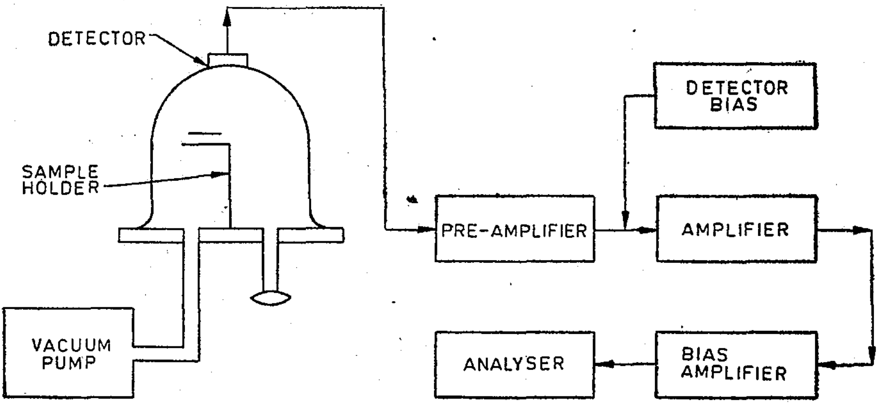

FIG. 2 SCHEMATIC DIAGRAM OF SURFACE BARRIER (SOLID STATE) ALPHA SPECTROMETER

3.1.5 The detector in this spectrometer arrangement is germanium or silicon solid state device which detects and produces different energy pulses depending upon impinging *alpha*-particles. Vacuum of the order of 1 kPa is created in the chamber where detector and sample are housed so that the presence of air does not attenuate *alpha*-particles. These pulses of different energy are processed by the preamplifier and finally fed to the analyser unit which sorts out the different pulses according to their energy. Thus *alpha*-particles of different energies coming from a given sample can be sorted out and counted.

3.2 Beta Counter

3.2.1 Beta emitting radionuclides are counted either by end window Geiger-Muller (GM) counter or by liquid scintillation counter depending on the energy of *beta* emitted. The GM counter is essentially an ionization chamber filled with argon gas along with a small amount of halogen gas acting as quencher and works at high voltage. The essential constituents of GM counter are GM tube, high voltage supply for the tube, a load resister and an electronic voltage pulse counter. The counter is set to accept only voltage pulses of the size produced by the Geiger tube. Since the pulses from Geiger tube are all of the same size regardless of the number of electron pairs produced, it cannot directly distinguish between *alpha*, *beta* and *gamma* radiation without the use of external absorber.

3.2.1.1 The Geiger tube should be operated at correct voltage within the plateau region. A typical graph showing range of the operation of various counters is given below:

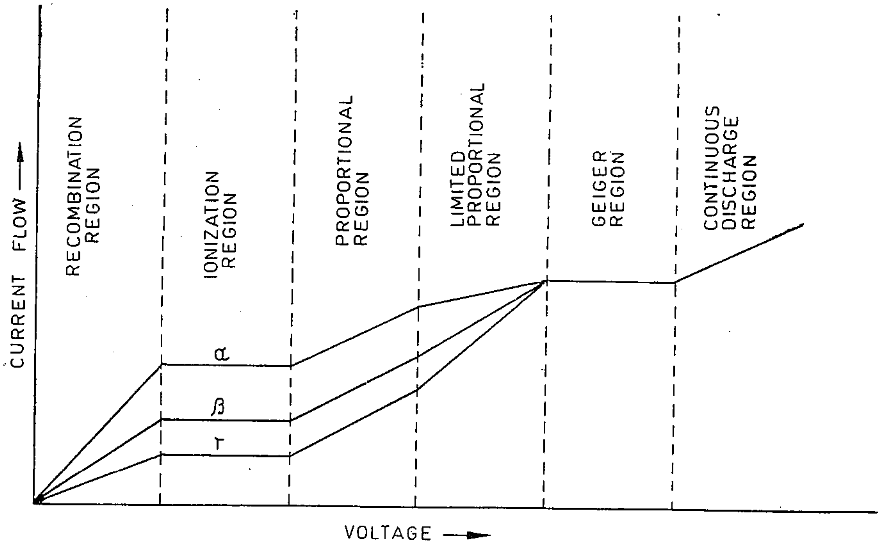

FIG. 3 RANGE OF OPERATION VOLTAGE OF VARIOUS COUNTERS

3.2.1.2 The operating voltage of Geiger tube is taken as the mid point of the plateau. If V_1 is the voltage where plateau starts and V_2 the higher value of plateau, the tube should be operated at $\frac{V_1 + V_2}{2}$ volts.

The fluctuation in the power supply does not affect the count rate at this operational voltage. Another method to find optimum voltage of the tube is to keep note down the background counts '*B*' and reference source counts '*S*' at regularly increasing voltage. The graph between S/B^2 versus voltage is plotted and the voltage for the maximum value of S/B^2 is chosen as the optimum voltage.

3.2.2 Low Background Beta Counter (Gas Flow Beta Counter) — This counter is essentially a GM counter with the only difference that instead of sealed gas in GM counter, the gas is in continuous flow in this counter. In addition to the main counter two more counters called guard counters (or umbrella counters) are used. The schematic diagram of the arrangement is given in Fig. 4.

3.2.2.1 Argon gas flowing through counters is bubbled through *iso*-propyl alcohol and allowed to escape through an oil bubbler. Any back ground radiation falling through guard counter produce pulses in the guard and main counters which are fed to coincidence analyser through paralysis time units as they will reject two pulses coming simultaneously thus reducing the background considerably. Only the single pulses produced by the sample in the main counter are registered in the scaler. Thus the pulses due to external background are filtered out. A comparison is shown below:

	Background	Efficiency, percent
GM Counter	8-15 cpm	10-15
Gas Flow Counter	~ 0·6 cpm	45

3.2.3 *Liquid Scintillation Counter* — The working of the counter is based on the measurement of light flashes produced by ionizing radiation. The light is seen by the photomultiplier tube, amplified, processed and the events are counted. This counter is used for measurement of weak energy *beta* emitters like, ^{3}H, ^{14}C, and ^{32}p. The sample is mixed with liquid scintillator in a glass vial of low potassium content and kept for counting. The liquid scintillator is a solution of primary and secondary fluorescent materials and naphthalene in 1, 4 dioxane. Different liquid scintillation cocktails may be used depending upon the requirement, for example, toluene or xylene based liquid scintillators. The counting efficiency varies from 20 to 50 percent for aqueous samples of different volumes. The composition of a typical liquid scintillator is given below:

Naphthalene	100 g
PPO (2, 5-diphenyl oxazole)	7·0 g
POPOP (2, 2-para phenylene-bis-5-phenyl oxazole)	0·35 g
1, 4-Dioxane	1 000 ml

3.2.3.1 A schematic diagram of liquid scintillation counter is shown in Fig. 5.

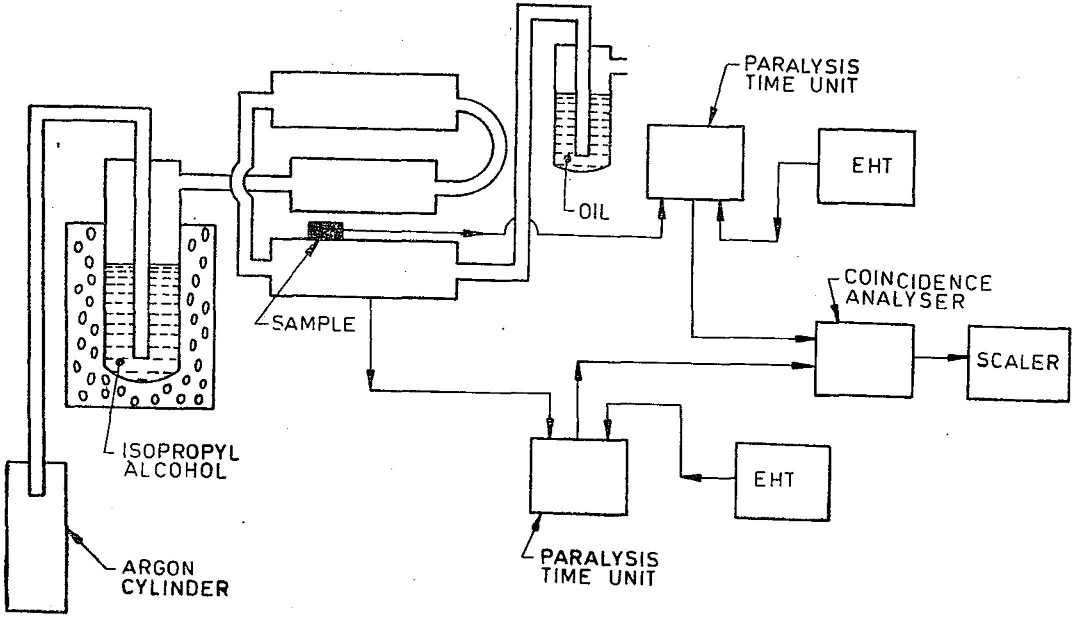

Fig. 4 Low Background β-Counter

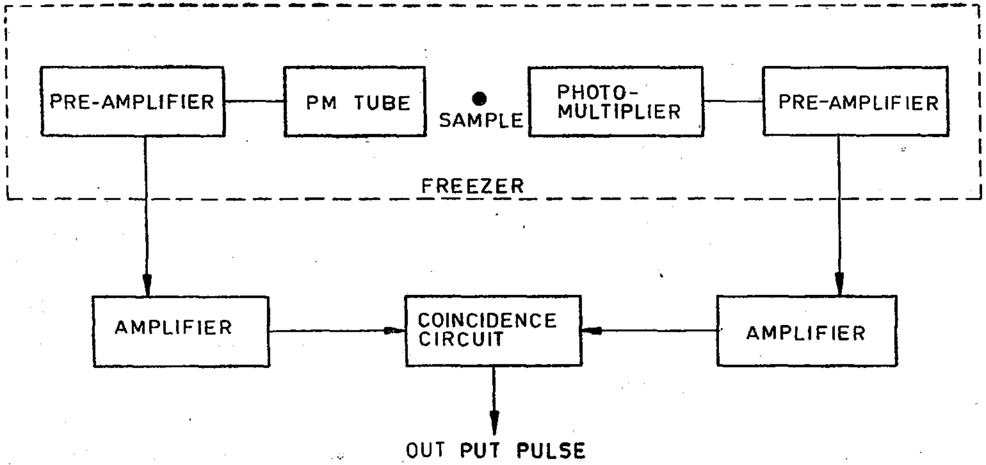

FIG. 5 SCHEMATIC DIAGRAM OF LIQUID SCINTILLATION COUNTER

3.3 Gamma Counter

3.3.1 The working of a *gamma* counter is based on scintillations produced by *gamma* rays. Thallium activated sodium iodide crystal is used as the detector for gamma rays. The scintillations produced in the crystals are seen by photomultiplier tube, amplified, processed and counted by the scaler. Usually the NaI (Tl) crystal is of well type in which the sample, in plastic vials, may be kept to get good counting geometry. If the sample is to be counted in bulk, a cylindrical crystal with plastic Marinelli beaker for keeping sample is employed. The *gamma* counting apparatus requires a good amount of lead shielding to reduce the background radiations. The crystal geometry is shown in Fig. 6 and schematic diagram of γ-counting apparatus is shown in Fig. 7.

3.4 Background and Efficiency

3.4.1 To use the equipment for measurement, it is necessary first to determine the count rate observed by itself because of natural background and electronic noise. The background is a built-in correction to the count rate observed when the sample is not mounted. If C_s is the count rate, that is, count per second of a sample and C_b is the count rate of background the net count rate of the sample is equal to $C_s - C_b$ counts/s.

3.4.2 The net count rate depends upon the efficiency of the counter and can differ from one instrument to the other. All the disintegrations emitted in unit time by a radionuclide are not registered in the counter and the count rate observed is about 25 to 40 percent of the disintegration rate. The disintegration rate is a true measure of the radioactivity content. It is therefore necessary to determine the efficiency of the counter, so that the count rate could be converted into the disintegration rate.

The efficiency is determined by counting a reference source containing known amount of radioelement. Natural uranium for alpha and ^{40}K for beta are used as reference sources. The disintegration rate of the reference source is calculated from the amount of radioelement present and the decay constant. Thus,

$$\frac{\text{Net count rate observed of the reference source}}{\text{Calculated disintegration rates of the reference source}} \times 100$$

3.4.3 Sometimes the count rate has to be corrected for loss of counts by absorption in the residue (high dissolved solids of a water sample), back scatter, etc. It is therefore better to check the overall process efficiency (chemical plus instrumental) by using a spike in the sample, and finding the count rate according to the procedure. A spike consists of a solution of the radioisotope of known radioactivity content.

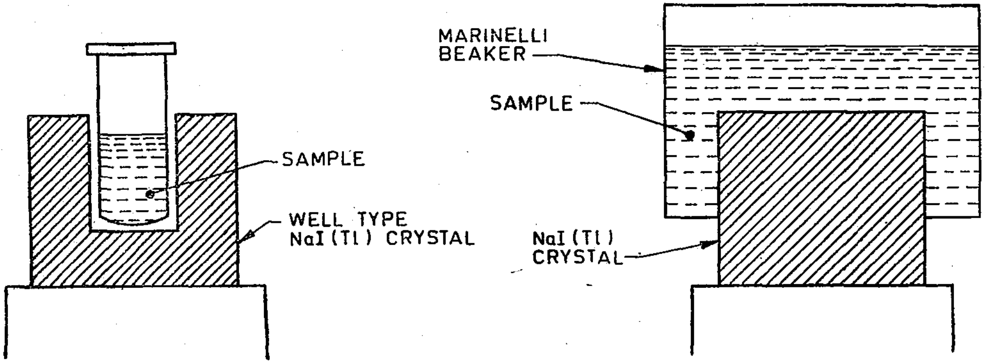

FIG. 6 CRYSTAL GEOMETRY

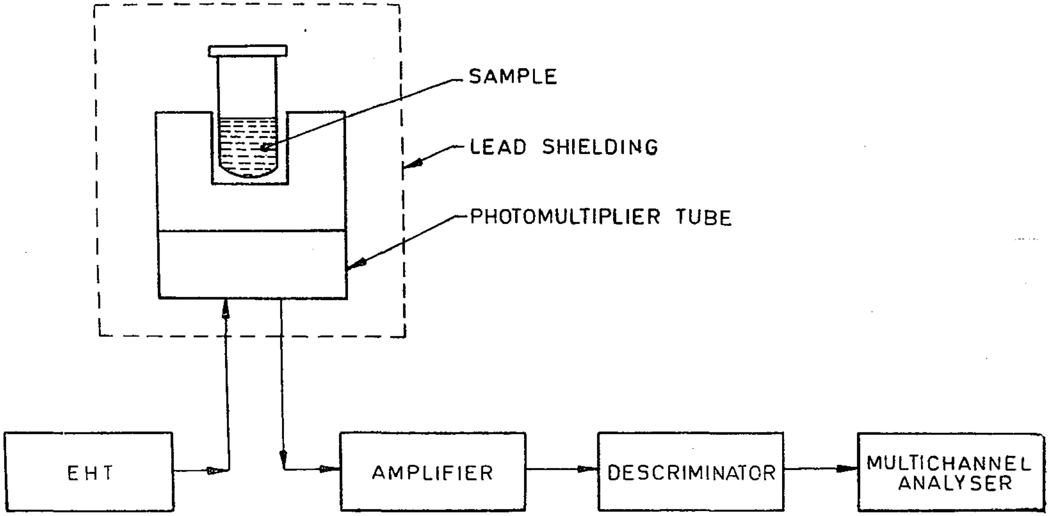

FIG. 7 SCHEMATIC DIAGRAM OF γ COUNTING APPARATUS

3.4.4 *Decay Correction* — It is not the practice to apply a decay correction for gross activity measurements. If the gross activity (α or $\beta - \gamma$) decay at a rapid rate, a radiochemical identification may have to be made.

3.5 Disintegration Rate and Units of Radioactivity

3.5.1 The disintegration rate is related to the conventional unit of measurement of radioactivity curie (C_i) as follows:

$$\text{One Curie} = 3{\cdot}7 \times 10^{10} \text{ disintegrations per second (dps) or}$$
$$= 2{\cdot}22 \times 10^{12} \text{ disintegrations per minute (dpm).}$$

The prefixes micro (10^{-6}), nano (10^{-9}) and pico (10^{-12}) are used for convenience when the activity levels are low. Thus one pico curie (pCi) = 2·22 dpm. The SI unit of measurement of radioactivity is called the bacquerel (Bq).

$$1 \text{ Bq} = 1 \text{ dps}$$

3.6 Statistics of Counting

3.6.1 Radioactivity decay is a random phenomenon and the counts registered are subject to a statistical fluctuation. For obtaining statistical reliability, a large number of counts have to be registered and several measurements carried out on the sample mount.

3.6.1.1 The count rates follow normal distribution. Therefore the standard deviation, which is a measure of fluctuation, may be calculated as follows:

$$\text{Standard deviations, } \sigma = \frac{1}{N-1}\left[\Sigma \left(C_s - C_b \right)^2\right]^{\frac{1}{2}}$$

where

N = number of time the sample is counted,

C_s = net count rate observed each time, and

C_b = arithmetic mean of all the count rates.

3.6.2 It is, however, not feasible to count each sample several number of times. For practical purposes the standard deviation (σ) may be determined by finding C_s in a single count. Ideally the background should be determined for the same period of time as that of the sample, but as this is not always possible, it is practical to determine the background counts for 60 minutes once daily. If the total count obtained for the sample is C_s in time t_s seconds, and for background C_b counts for t_b seconds the net count rate is given by the following expression:

$$\text{Net count rate} = \frac{C_s}{t_s} - \frac{C_b}{t_b}$$

Applying the standard deviation correction for sample and background counts we get

$$\sigma b = \frac{C_{\mathrm{b}}^{\frac{1}{2}}}{t_{\mathrm{b}}},\ \sigma_{\mathrm{s}} = \frac{C_{\mathrm{s}}^{\frac{1}{2}}}{t_{\mathrm{s}}} \text{ and}$$

$$\sigma \text{ net} = [(\ \sigma_{\mathrm{b}}\ )^2 + (\ \sigma_{\mathrm{s}}\ )^2]^{1/2}$$

The count rate of a sample is therefore:

$$\left[\frac{C_{\mathrm{s}}}{t_{\mathrm{s}}} - \frac{C_{\mathrm{b}}}{t_{\mathrm{b}}}\right] \pm \sigma \text{ net}$$

The disintegration rate of the sample is reported as follows:

Activity in Bq/litre =

$$\left[\frac{C_{\mathrm{s}}}{t_{\mathrm{s}}} - \frac{C_{\mathrm{b}}}{t_{\mathrm{b}}}\right] \pm \sigma \text{ net} \times \frac{100}{E} \times \frac{1}{V}$$

where

E = efficiency of the counter, percent; and

V = volume of sample, litres.

3.6.3 The value of the standard deviation (σ) is useful to calculate the percentage error and the confidence level of counting.

$$\text{Percentage error} = \frac{\sigma \times 100}{\text{net count rate}}$$

3.6.3.1 Considering the areas occupied in the normal distribution diagram the results may be stated at different levels of confidence as follows:

Net count rate ± 1·00 σ = 68 percent confidence

Net count rate ± 1·65 σ = 90 percent confidence

Net count rate ± 2·00 σ = 95 percent confidence

Net count rate ± 3·00 σ = 100 percent confidence

3.6.3.2 Thus, the net count rate of a sample ± one σ implies that the result can be taken with confidence that the observed count rate will fall within the range as much as 68 times out-of 100 observations.

4. PROCEDURE

4.1 *Beta* Reference Source — *Beta*-standard is prepared from ^{40}K radionuclide. Natural potassium contains 0·011 7 percent of ^{40}K with half life of 1·28 × 10^9 years. This radioisotope emits 89·3 percent *beta*, its energy being 1·325 Mev. The specific activity of potassium chloride is given below:

$$\text{Specific activity} = \frac{6{\cdot}023 \times 10^{23}}{40} \times \frac{39{\cdot}102}{74{\cdot}555} \times \frac{0{\cdot}011\ 7}{100}$$

$$\times \frac{0{\cdot}693}{1{\cdot}28 \times 10^9 \times 365{\cdot}25 \times 24 \times 60} \times \frac{89{\cdot}33}{100} = 849{\cdot}63 \text{ dpm/g}$$

4.1.1 Take analytical reagent grade potassium chloride and warm it in an oven at 105°C. Grind thoroughly and pass through 200 mesh. Weigh a clean aluminium planchet and add about 100 mg of powdered potassium chloride. Find its exact mass and tap gently to spread the powder evenly. Add about 0·5 ml 10 percent collodian solution to cover the powder completely and evenly. Dry first in air and then under an infrared lamp. Calculate the dps of the source and note the value at the bottom of the planchet. (100 mg of KCl = 1·41 dps).

4.1.2 Alternate reference sources may be prepared similarly. If *alpha* contamination is expected in the source, the same may be avoided by covering with thin aluminium foil (7 mg/cm^2). The selected radionuclide for reference standard should be of fairly high radioactive half life otherwise decay correction shall have to be applied.

4.2 Alpha Reference Source — The reference standard is prepared either from natural uranium or plutonium. These have high radioactive half life and are easy to electroplate on the stainless steel disc.

4.2.1 Prepare uranylnitrate solution of concentration 100 $\mu g/ml$ (natural uranium). Transfer 2 ml solution into a 10 ml beaker. Evaporate to dryness under infrared lamp. Dissolve the residue in 0·2 to 0·3 ml of 0·2 N sulphuric acid. Transfer the solution into electro-deposition cell with 10 ml of 4N ammonium sulphate. The electrodeposition cell is made of either polytetra fluoro ethylene (PTFE) or polymethyl methacrylate (PMA) tube of 2 cm internal diameter and 8 cm length screwed to a stainless steel cup. Place stainless steel disc in the cup and screw PTFE/PMA tube liquid-liquid tight. A platinum wire serves as anode while the stainless steel disc works as cathode. Stir the solution constantly with anode itself serving as the stirrer. Adjust the *p*H to 2·5 with 1 N sulphuric acid. Connect the cell to the D.C. circuit at 5 volt and 300 mA current. Pass the current for about 2 hours. Add 1 ml ammonia, continue stirring for a minute and then disconnect the cell and wash the disc with distilled water and then with alcohol. Dry under infrared lamp and flame over the burner. Cool and preserve safely.

4.2.2 Transfer the cell solution and the washings in a beaker and determine the uranium content by fluorometry. From the difference in uranium content in solution before and after electrodepositions calculate the amount of uranium deposited on the stainless steel disc. Compute the dpm of the standard thus prepared given by the equation.

1 μg uramium (natural) = 1·524 dpm.

(Based on following data):

Isotope	*Abundance, Percent*	*Half Life, Years*
U-238	99·275	$4{\cdot}468\,324 \times 10^9$
U-235	0·72	$7{\cdot}038\,5 \times 10^8$
U-234	0·005 4	$2{\cdot}446 \times 10^5$

The reference source of other nuclides may be prepared similarly depending on the requirement of alpha energy.

4.3 Preparation of Carriers — The number of atoms/molecules of radio-active elements encountered in drinking water and also in waste waters are too small to separate from the bulk samples. A solution of a stable isotope of the radionuclide of interest is added prior to the precipitation. Thus along with the stable isotope, the radionuclide also gets precipitated. The solutions which enhance the precipitation of radionuclides are called 'carriers'. The carriers are essential for radiochemical separation and estimation of radionuclides, and are normally prepared in the same form as that of expected radionuclide. The carriers are prepared by dissolving known amounts of water soluble salts and are expressed as mg/ml. The carrier solutions thus prepared are standardized by gravimetric methods to know the exact strength as it is taken into account for calculating the percentage recovery. Typical example of preparing and standardising barium carrier is given below.

4.3.1 To prepare 5 mg/ml barium carrier take 0·951 6 g of analytical reagent grade bariumnitrate [$Ba(NO_3)_2$] into a 100 ml standard flask and make up to the mark with distilled water. This mass of barium nitrate for 100 ml solution is arrived at by using the following factor:

$$\frac{261{\cdot}38}{137{\cdot}34} \times 5 \times 10^{-3} \times 100$$

For standardization carry out precipitation under conditions similar to those employed in the radiochemical analysis of the sample. Place in triplicate 3 ml aliquots of the barium carrier solution in the 40 ml centrifuge tubes. Acidify with dilute nitric acid and dilute each to about 20 ml with distilled water and heat in water bath for about 3 minutes. Add 1 ml of 3 M sulphuric acid dropwise while shaking the centrifuge tube. Filter through weighed sintered crucible and wash the barium sulphate precipitate three times with hot water containing few drops of sulphuric acid followed by 3 ml of ethyl alcohol. Dry the sintered crucible by solution and then at 110°C for 20 minutes. Cool in a desiccator and weigh. Repeat drying till a constant mass is obtained.

$$\text{Concentration of } Ba^{+2} \text{ mg/ml} = \frac{M \times 0{\cdot}589}{3}$$

where

M = mass of $BaSO_4$, mg.

4.3.2 The carriers of other elements may be prepared and standardized in a similar manner. Some of the carriers routinely required are given below.

4.3.2.1 *Bismuth — 100 mg/ml* — Dissolve 23·212 0 g bismuth contents [Bi $(NO_3)_3.5H_2O$] in 63 ml concentrated nitric acid and dilute to 100 ml with distilled water.

4.3.2.2 *Calcium — 200 mg/ml* — Dissolve 73·368 0 g calcium chloride ($CaCl_2.2H_2O$) in distilled water and make up to 100 ml or treat 49·904 0 g $CaCO_3$ with sufficient amount of hydrochloric acid slowly till the carbonate is fully decomposed, and make up to 100 ml by distilled water.

4.3.2.3 *Cesium — 10 mg/ml* — Dissolve 1·466 6 g cesium nitrate [Cs (NO_3)] in distilled water and make up to 100 ml.

4.3.2.4 *Cobalt — 100 mg/ml* — Dissolve 28·050 0 g cobalt chloride ($CoCl_3$) in distilled water and make up to 100 ml.

4.3.2.5 *Iodide — 10 mg/ml* — Dissolve 1·307 5 g potassium iodide (KI) in distilled water, and make up to 100 ml.

4.3.2.6 *Iron — 10 mg/ml* — Dissolve 2·904 7 g ferric chloride ($FeCl_3$) in distilled water and make up to 100 ml.

4.3.2.7 *Lanthanum — 10 mg/ml* — Dissolve 2·345 6 g lanthanum oxide (La_2O_3) in minimum quantity of concentrated HNO_3 and make up to 100 ml by distilled water.

4.3.2.8 *Lead — 100 mg/ml* — Dissolve 15·986 0 g lead nitrate [Pb $(NO_3)_2$] in distilled water and make up to 100 ml.

4.3.2.9 *Nickel — 10 mg/ml* — Dissolve 4·953 5 g nickel nitrate [Ni $(NO_3)_2 \cdot 6H_2O$] in distilled water and make up to 100 ml.

4.3.2.10 *Ruthenium — 10 mg/ml* — Dissolve 3·910 3 g ruthenium nitrate [Ru $(NO_3)_3 \cdot 6H_2O$] in distilled water and make up to 100 ml.

4.3.2.11 *Strontium — 10 mg/ml* — Dissolve 3·237 9 g stronium nitrate [Sr $(NO_3)_2 \cdot 4H_2O$] in distilled water and make up to 100 ml.

4.3.2.12 *Yitrium — 10 mg/ml* — Dissolve 4·307 7 g yitrium nitrate [Y $(NO_3)_3 \cdot 6H_2O$] in distilled water and make up to 100 ml.

4.3.2.13 *Zinc — 10 mg/ml* — Dissolve 4·550 8 g zinc nitrate [Zn $(NO_3)_2.6H_2O$] in distilled water and make up to 100 ml.

4.3.2.14 *Zirconium — 10 mg/ml* — Dissolve 4·706 5 g zirconium nitrate [Zn $(NO_3)_2.5H_2O$] in distilled water and make up to 100 ml.

4.4 Gross Activity Measurements

4.4.1 It is always necessary to know the total dissolved solids (TDS) in water and waste waters before starting gross measurements. The amount of sample to be evaporated and plancheted depends on the TDS as high TDS will absorb alphas in it. The requirements for the gross measurements are given in Table 1.

TABLE 1 REQUIREMENTS FOR GROSS MEASUREMENTS

Sl No.	Description	Requirements	
		Alpha	Beta
(1)	(2)	(3)	(4)
i)	Planchet size, shape and material	2·5 cm or 5·0 cm diameter rimless stainless steel disc	2·5 cm diameter, 0·3 cm depth aluminium dish
ii)	Residue spread on the planchet	1 mg/cm^2 (5 mg)	20 mg/cm^2 (100 mg)
iii)	Volume of sample plancheted	0·5 to 1·0 ml	0·5 ml to 1·0 ml
iv)	Background counts	0·5 to 1·0 cpm	5 to 12 cpm
v)	Counting time	100 to 400 minutes (depend on count rate)	100 minutes (depend on count rate)
vi)	Efficiency	25 percent	10 to 18 percent
vii)	Reference source	U material electroplated	40 K
viii)	Counting device	Zinc sulphide (ZnS) Silver (Ag)	End window
		Scintillation counter	GM counter

4.4.2 The gross measurements indicate the quality of water and waste waters. The following guidelines are suggested for further analysis of drinking water:

Gross alpha activity < 0·1 Bq/l — No further examination is essential

Gross alpha activity > 0·1 Bq/l — Further examination for individual radionuclides is essential

Gross beta activity < 0·8 Bq/l — No further examination is essential

0·8 Bq/l > (Gross beta activity — 40_K activity) — No further examination is essential

(Gross activity — 40_K activity) >0·8 Bq/l — Further examination for individual radionuclides is essential

4.4.3 Filter water/waste water sample through a Whatman No. 42 filter paper. Evaporate a known amount of an aliquot of the sample in a beaker under an infra red lamp (solids 5·0 mg for *alpha* and 100 mg for *beta*). When the solids start separating, transfer the slurry with a dropper into a stainless steel planchet of diameter 2·5 cm. Dry under infra-red lamp and count *alpha* in ZnS (Ag) scintillation counter.

$$\text{Gross alpha, Bq/l} = \frac{1\,000\,S\,(C_s - C_b)}{V\,(C_r - C_b)}$$

where

C_s = counts per second for sample,

C_r = counts per second for reference source,

S = disintegration per second for reference source,

C_b = background counts per second, and

V = volume in litres taken for evaporation.

4.4.4 For gross *beta* planchet evaporate sample in an aluminium dish, dry under an infra-red lamp and count *beta* using an end window GM counter. The *alpha*-interference in the *beta*-counting may be avoided by covering the sample with suitable aluminium foil. No *gamma*-interference is possible as the counter efficiency for *gamma* is very low.

The gross measurements of waste waters require special attention. These may have higher TDS. The waste water sample may either be diltuted to reduce TDS, or digested with nitric acid. In case dilution or digestion with nitric acid does not help an alternate procedure to separate gross activity as calcium phosphate [$Ca_3(PO_4)_2$] may be followed. The procedure is given in **4.4.4.1**.

4.4.4.1 To the sample add 100 mg calcium carrier and 10 ml 1 M sodium phosphate (Na_2HPO_4) and make alkaline with 1 : 1 ammonia. Discard supernate. Dissolve the precipitate in 1·0 ml 8 M nitric acid. Transfer into a 5 ml standard flask and make up to mark. Planchet 1 0 ml on stainless steel planchet of 2·5 cm diameter, dry under lamp and count beta. This beta, along with cesium and iodine beta gives total gross beta. Dilute remaining 4 ml solution to about 10 ml. Add about 5 ml 1·5 M hydroxyl amine hydrochloride ($NH_2OH.HCl$) solution, heat and add 100 mg bismuth carrier and 5 ml 1 M phosphoric acid. Cool and centrifuge. Supernate contains uranium and radium.

4.4.4.2 To the precipitate add 1 N hydrochloric acid till the precipitate dissolves. Add 2 mg lanthanum carrier. Precipitate (LaF_3) with HF and wash the precipitate with hydrochloric acid-hydrofluoric acid (HCl — HF) and nitric acid-hydrofluoric acid (HNO_3 — HF) wash solutions followed by distilled water. Planchet precipitate on stainless steel disc, dry under lamp, fire and count *alpha* (Th, Pu, transuranica).

Add the *alpha* activity due to uranium and radium from the supernatant to get the total gross *alpha* activity.

4.5 Analysis of Individual Radionuclides

4.5.0 The amount of water to be sampled depends on the location and quality of water. If the radioactive contamination from man-made sources is not expected, the level of radioactivity can be expected to be low and a sufficiently large amount of sample is required. Ten to fifteen litres of sample is needed for the estimation of one radionuclide alone. Although in principle the radionuclides may be determined separately, taking fresh sample for each radionuclide will require a large quantity of sample. Thus radiochemical analysis using the same sample should be preferred for determination of various nuclides.

4.5.1 *Iodine 131*

4.5.1.1 *Principle* — Iodine is precipitated from the sample as silver iodide by exchanging with silver chloride. The silver iodide is further purified by use of ziroconium carrier and precipitating the carrier as ziroconium hydroxide. The purified silver iodide precipitate is washed, dried and counted.

4.5.1.2 *Reagents*

a) *Iodide carrier* — 10 mg/ml,

b) *Zirconium carrier* — 10 mg/ml,

c) *Hydroxylamine hydrochloride* ($NH_2OH.HCl$) — solid,

d) *Silver chloride* (AgCl) — solid,

e) *Nitric acid* (HNO_3) — 2N,

f) *Potassium cyanide solution* — 2 percent, and

g) *Ammonia solution* (NH_4OH) — 6 N.

4.5.1.3 *Procedure* — Filter 5 litres of sample water through Whatman filter paper No. 42. Take 4 litres of filtered sample in a 5 litre beaker and add 1 ml iodide carrier and 1·0 g of $NH_2OH.HCl$ to reduce any iodate or iodine to iodide. Add 10 ml 2H HNO_3 and 200 mg AgCl. Stir for about 30 minutes and allow to settle for 3 to 4 hours. Decant and centrifuge the slurry. Preserve the supernate and washings for strontium and cesium analysis. Add 10 ml of 6 N NH_4OH to the precipitate to dissolve excess AgCl. Centrifuge and separate AgCl precipitate. Wash the precipitate in 5 ml of 2 percent potassium cyanide solution. Add 1 ml zirconium carrier and precipitate $Zr(OH)_4$ by adding dropwise 6 N NH_4OH solution. Centrifuge and separate $Zr(OH)_4$. To the supernate add drop by drop 2 N nitric acid till silver iodide (AgI) is precipitated. Centrifuge and wash the precipitate with 2 N HNO_3. Redissolve the AgI precipitate in 5 ml of 2 percent KCN solution.

Centrifuge if there is any residue. In the supernate again precipitate AgI with 2N HNO_3 and wash it with distilled water. Transfer the AgI precipitate on a washed, dried and pre-weighed filter paper disc (surface area 2·5 cm^3, Whatman filter No. 542). Wash the precipitate on the filter paper disc with pure ethyl alcohol and dry at 100°C for 5 minutes.

Weigh the precipitate and keep it along with the paper disc in an aluminium planchet. Count beta (β) due to 131 I using a GM counter. Using the same counter take background counts of an empty aluminium planchet. Count the reference standard also for converting counts per sec into disintegrations per sec.

4.5.1.4 *Calculation* — Calculate the concentration of iodine activity in bacquerel/litre (Bq/l) as follows:

$$\text{Iodine activity (Bq/l)} = \left[\left(\frac{C_s}{t_s} - \frac{C_b}{t_b}\right) \pm \sqrt{\frac{C_s}{t_s^2} + \frac{C_b}{t_b^2}}\,\right] \times \frac{S}{\left(\dfrac{C_{ref}}{t_r} - \dfrac{C_b}{t_b}\right)} \times \frac{100}{y} \times \frac{1}{V} \times e^{\frac{0{\cdot}693\,t}{t_{\frac{1}{2}}}}$$

where

C_b = background counts in t_b seconds,

C_s = sample counts in t_s seconds,

C_{ref} = reference standard counts in t_r seconds,

S = reference standard strength in disintegration per second,

y = percent chemical recovery,

V = volume of sample taken in litres,

t = time elapsed in hours between sampling and counting, and

$t_{\frac{1}{2}}$ = half life of 131 I in hours (8·06 days).

$$y \text{ (Percent chemical recovery)} = \frac{\text{mass of AgI precipitate in mg} \times 126{\cdot}9}{\text{mass of carrier in mg} \times 234{\cdot}8} \times 100$$

4.5.2 *Strontium 89 and 90*

4.5.2.1 *Principle* — Strontium is precipitated as strontium carbonate and purified from other interfering radionuclides. It is finally counted as build-up of yttrium-90 for 90_{sr} and as strontium carbonate for 89_{sr} and 90_{sr}.

4.5.2.2 *Reagents*

a) *Fuming nitric acid* — 6 N and 1 N nitric acid,

b) *Hydrochloric acid* — 11 N.

c) *Ammonium carbonate* (NH_4)$_2CO_3.H_2O$ — saturated solution,
d) *Barium carrier* — 10 mg/ml,
e) *Cesium carrier* — 10 mg/ml,
f) *Iron carrier* — 10 mg/ml,
g) *Strontium carrier* — 10 mg/ml,
h) *Yttrium carrier* — 10 mg/ml,
j) *Sodium carbonate* Na_2CO_3 — saturated solution,
k) *Ammonium oxalate* (NH_4)$_2C_2O_4.H_2O$ — saturated solution,
m) *Sodium chromate* ($Na_2CrO_4.H_2O$) — 5 percent solution,
n) *Acetic acid* — ($CH_3CO\ OH$) — 6 M,
p) *Ammonium acetate* — ($CH_3COO\ NH_4$) — 6 M, and
q) *Ammonium solution* — (NH_4OH) — concentrated (1:1).

4.5.2.3 *Procedure* — Take 4 litres of filtered sample of the supernate after iodine separation and acidify with 50 ml concentrate HCl. Add 6 ml strontium carrier, 4 ml cesium carrier and raise the *p*H to 9 with concentrated NH_4OH.. Check with *p*H meter. Heat the solution to about 80°C and add slowly saturated solution of ammonium carbonate. Allow the precipitate to settle overnight. Decant the supernate and centrifuge the slurry. Wash the precipitate by distilled water and add the washings to the supernate. Keep the total supernate for cesium — 137 analysis. Dissolve the precipitate in minimum volume of 6 N nitric acid and evaporate to dryness. Dissolve the residue in 6 N nitric acid and cool in an ice bath for 10 minutes. Add fuming nitric acid to precipitate strontium nitrate. Keep for about 15 minutes. Centrifuge and discard the supernate. Once again dissolve the residue in 6 N nitric acid and cool in ice bath for 10 minutes. Add fuming nitric acid to precipitate strontium nitrate. Keep for about 15 minutes, centrifuge and discard the supernate. Dissolve the precipitate in 2 to 3 ml distilled water and add a drop of methyl red indicator. Neutralize with ammonium hydroxide (1:1) (colour change red to yellow). Add 2 ml 6 M acetic acid and 4 ml 6 M ammonium acetate solution. Add 0·5 ml barium carrier (10 mg/ml) and precipitate barium chromate with 2 ml of 5 percent sodium chromate solution. Centrifuge and collect the supernate and a distilled water washing in another centrifuge tube. Precipitate strontium carbonate with 2 ml saturated sodium carbonate solution. Centrifuge and discard the supernate. Dissolve the precipitate slowly in 2 ml of 1 N nitric acid. Add 0·5 ml iron carrier (10 mg/ml). Add 2 to 3 ml 1:1 ammonium hydroxide to precipitate ferric hydroxide. Centrifuge and discard the precipitate. To the supernate add 2 to 3 ml saturated sodium carbonate solution to precipitate strontium carbonate again. Centrifuge and discard the supernate. Dissolve the strontium carbonate precipitate in 1-2 ml 1 N nitric acid. Add 0·5 ml yttrium carrier (10 mg/ml) and keep the solution for 15 days for 90 Y to grow. After an equilibrium between 90 S_r and 90 Y is reached (15 days), dissolve the residue in 1 to 2 ml 1 N nitric acid and precipitate $Y(OH)_3$ with 1:1 ammonium hydroxide. Centrifuge and collect the supernate along with the washings for strontium 89 + 90. Dissolve the $Y(OH)_3$ precipitate in 1 ml 1 N nitric acid and add 5 ml saturated ammonium oxalate solution to precipitate yttrium oxalate. Wash the precipitate twice with distilled water and transfer it with water on a pre-weighed aluminium planchet. Dry the precipitate in aluminium planchet under an infra-red lamp. Count beta (β) due to strontium-90 activity using a G.M. Counter (A). Using the same counter take back ground counts of ash washed empty aluminium planchet. Count the reference standard also for converting counts/sec into disintegration/sec. To the supernate and washings add saturated sodium carbonate solution to precipitate strontium carbonate. Centrifuge and wash the precipitate with distilled water. Transfer the precipitate in a pre-weighed aluminium planchet. Dry the precipitate under an infra-red lamp. Count beta (β) by using G.M. Counter to get activity due to strontium 89 + 90 (B). Evaluate 89_{sr} from the difference (B-A).

4.5.2.4 *Calculations* — Calculate the concentration of active strontium in bacquerel per litre (Bq/l) as follows:

$$\text{Strontium activity (Bq/l)} = \left[\left(\frac{C_s}{t_s} - \frac{C_b}{t_b}\right) + \sqrt{\frac{C_s}{t_s^2} + \frac{C_b}{t_b^2}}\right] \times \left[\frac{S}{\left(\frac{C_{ref}}{t_r} - \frac{C_b}{t_b}\right)} \times \frac{100}{y} \times \frac{1}{V}\right]$$

where the different terms have the same meanings as in **4.5.1.4**.

Percent chemical recovery y for 90_{sr} =

$$\frac{\text{Mass of } Y_2(C_2O_4)_3\, 9H_2O \text{ precipitate in mg} \times \frac{177{\cdot}8}{603{\cdot}8} \times 100}{\text{Mass of yttrium carrier taken in mg}}$$

Percent chemical recovery y for Sr 89 + 90 =

$$\frac{\text{Mass of Sr } CO_3 \text{ precipitate in mg} \times \frac{87{\cdot}6}{147{\cdot}6} \times 100}{\text{Mass of Sr carrier taken in mg}}$$

4.5.3 *Cesium 134 and 137*

4.5.3.1 *Cesium* 134 and 137 is coprecipitated under acidic condition with ammonium molybdophosphate. It is purified using ion exchange column and finally percipitated as Cesium-stannic chloride and counted for β (beta).

4.5.3.2 *Reagents*

- a) *Ammonium nitrate* (NH_4NO_3) — solid,
- b) *Ammonium molybdophosphate* (NH_4)$_3$ P (Mo_3O_{10})$_4$ — solid,
- c) *Cesium carrier* — 10 mg/ml,
- d) *Hydrochloric acid* — 6 M,
- e) *Nitric Acid* — concentrated,
- f) *Ammonia solution* — 1 N,
- g) *Stannic chloride* ($Sn\ Cl_4$) — 1 N, and
- h) *Strong cation-exchange resin.*

NOTE — Dowex — 50 (50 to 100 mesh) has been found suitable.

4.5.3.3 *Procedure* — Take 4 litres of filtered sample or the supernate after strontium separation. Bring down the *p*H of the solution to 1·0 by using concentrated nitric acid. Add about 8 g of ammonium nitrate and about 3 g of ammonium nitrate and about 3 g of ammonium molybdophosphate. Stir mechanically for about 30 minutes. Allow to settle overnight. Decant the supernate and centrifuge the slurry. Dissolve the precipitate in 1 N ammonium hydroxide. Prepare a cation exchange resin column (about 5 to 8 cm resin height in about 1 cm diameter glass column). Condition it with 20 to 30 ml of ammonium hydroxide solution by passing at a rate of 2 to 3 ml per minute for 10 minutes. Pass the solution containing the dissolved precipitate through the column slowly (flow rate about 6 drops/min). Wash the column with about 50 to 60 ml 1 N ammonium hydroxide, following by distilled water till the effluent is free of ammonia (no ammonia smell). Elute the column with about 70 ml of 6 N hydrochloric acid at a rate of 1 ml/min. Evaporate the eluate to dryness with about 5 ml concentrated nitric acid to sublime off any ammonia present. To the residue add 5 ml 6 N hydrochloric acid and an equal volume of ethyl alcohol. Add 5 ml 1 N $SnCl_4$ to precipitate to $Cs_2\ Sn\ Cl_6$. Wash the precipitate once with 6 N hydrochloric acid followed by the ethyl alcohol (distilled) washes Transfer the washed precipitate into a preweighed aluminium planchet. Dry the precipitate on the planchet under an infra-red lamp. Count beta (β) due to Cesium 134 + 137 activity using a G.M. Counter. Using the same counter take background counts of a washed empty aluminium planchet. Count the reference standard also for converting counts/sec into disintegrations/sec.

4.5.3.4 *Calculations* — Calculate the concentration of active cesium (Ce 134 + 137) in Bacquerel/litre (Bq/l) as follows:

$$\text{Cesium activity (Bq/l)} = \left[\left(\frac{C_s}{t_s} - \frac{C_b}{t_b} \pm \sqrt{\frac{C_s}{t_s^2} + \frac{C_b}{t_b^2}} \right) \right]$$

$$\times \left[\frac{S}{\left(\frac{C_{\text{ref}}}{t_{\text{r}}} - \frac{C_{\text{b}}}{t_{\text{b}}} \right)} \times \frac{100}{Y} \times \frac{1}{V} \right]$$

where the different terms have the usual meanings as in **4.5.1.4.**

$$\text{Percent chemical recovery, } Y = \frac{\text{Mass of } Cs_2\,SnCl_6 \text{ ppt in mg} \times \frac{265{\cdot}8}{597{\cdot}2} \times 100}{\text{Mass of cesium carrier taken in mg}}$$

NOTE — In case determination of separate concentration of Cesium 134 and Cesium 137 is required, take the *gamma* spectrum and evaluate ^{137}Cs and ^{134}Cs, by measuring area under the individual peaks in the spectrum and comparing with the standards.

4.5.4 *Sequential Separation of Radium, Thorium, Uranium, Plutonium and Americium* — Owing to the low concentration of the above radionuclides generally found in water samples, it is usually preferred to conduct sequential analysis from a single sample for these nuclides. However, these radionuclides may also be analysed individually. It should, however, be borne in mind that collection of a large amount of sample will be necessary if individual analysis of these nuclides is done.

4.5.4.1 *Principle of separation* — The radionuclides are separated from each other by taking advantage of their differing affinites for adsorption on cation and anion exchange resin columns in different media.

4.5.4.2 *Reagents*

a) *Nitric acid* — concentrated.
b) *Dilute nitric acid* (HNO_3) — 8N,
c) *Hydrochloric acid* (HCl) — 6 N, 0·75 N, 11 N, 0·2 N,
d) *Hydroxylamine hydrochloride* (NH_2OH — HCl) — solid
e) *Hydroxylamine hydrochloride* — 1·5 M solution,
f) *Phosphoric acid* — concentrated,
g) *Ammonia* (NH_4OH) *solution* — concentrated;
h) *Iron carrier* — 10 mg/l,
j) *Bismuth carrier* — 100 mg/ml,
k) *Calcium carrier* — 10 mg/ml,
m) *Plutonium tracer* — 1 to 10 dpm of $^{242}Pu/^{236}Pu$,
n) *Americium tracer* — 1 to 10 dpm of ^{243}Am,
p) *Strong cation exchanger* — 300 to 150 μm (50 to 100 mesh), and
q) *Strong anion exchanger* — 300 to 150 μm (50 to 100 mesh).

4.5.4.3 *Procedure* — Take 4 liters of filtered water sample. Acidify with about 50 ml concentrated nitric acid. Add 0·5 ml plutonium tracer (1 to 10 dpm). Add 0·2 ml Americium tracer about the same activity. Add 2·0 g solid hydroxylamine hydrochloride. Stir well and keep for 5 to 6 hours. Add 10 ml of iron carrier (10 mg/ml). Precipitate ferric hydroxide with concentrated ammonia, stirring continuously during addition. Keep overnight, decant and centrifuge the slurry. In the supernate again add concentrated ammonia, stirring continuously, keep overnight, decant and centrifuge. Combine the two precipitates of ferric hydroxide containing plutonium and americium. Preserve the supernates containing radium, thorium and uranium (S_1). Dissolve the precipitate in 3 to 4 ml dilute nitric acid (8 N) and make the volume to about 1 litre (*p*H about 2·0). Add 1 g hydroxylamine hydrochloride to reduce the plutonium to trivalent state. Heat the solution and add 1 ml bismuth carrier (100 mg/ml) followed by the addition of concentrated phosphoric acid slowly in hot solution. Keep it overnight. Decant, centrifuge and collect the precipitate. Preserve the supernate (S_2) which contains thorium in part, and dissolve the precipitate in 1 ml of 6 N hydrochloric acid. Add 10 ml of 0·2 N hydrochloric acid so as to get 0·75 N hydrochloric acid in solution. Centrifuge and discard the residue, if present. After preparing strong cation exchange resin column (5 to 8 cm resin height in about 1 cm diameter glass column) and condition the column with 20 to 30 ml 0·75 N hydrochloric acid at a rate of 2 to 3 ml/min for 10 minutes, pass the solution through the column at a rate of about 6 to 7 drops (about 0·5 ml) per minute. Wash the column with about 20 ml of 0·75 N hydrochloric acid. The column will return plutonium, americium and part of thorium. Elute the column with 25 ml concentrated hydrochloric acid at a rate of about 0·5 ml/minute. Evaporate the eluate to dryness after adding a few drops of concentrated nitric acid. Dissolve the residue in 15 ml 8 N nitric acid. After preparing a strong anion exchange resin column (about 5 to 8 cm resin height in about 1 cm diameter glass column) and conditioning the column with 20 to 30 ml 8 N nitric acid at a rate of 2 to 3 ml/min for 10 minutes, pass the solution through the column at a rate of about 0·5 ml/min. Wash the column with about 20 ml of 8 N nitric acid. Collect the effluent (F_1). Americium will pass through, while remaining thorium and plutonium along with small amount of residual iron present will be retained on the column. Pass 20 ml of concentrated hydrochloric acid to elute thorium from the column. Preserve the eluate (S_3). Elute the adsorbed plutonium with about 15 ml of 1·5 N hydroxylamine hydrochloride solution at a rate of 0·5 ml/minute. Evaporate the eluate containing plutonium to dryness. Dissolve the residue in concentrated nitric acid. (Add small instalments of about 2 to 3 ml each time). Finally evaporate the solution to dryness. Preserve the residue for carrying out plutonium determination by electroplating. Evaporate the effluent F_1 containing mainly americium and small amount of iron impurity to dryness and redissolve in concentrated hydrochloric acid. Pass this solution through a strong anion exchange resin column preconditioned with concentrated hydrochloric acid at a rate of about 0·5 ml/min to purify americium from iron impurity. Americium will pass through and iron will be retained in the column. Evaporate the effluent to dryness. Preserve the residue for carrying out determination of americium by electroplating. Combine the supernates S_1 and S_2 with the eluate S_3. Add 20 ml of calcium carrier (10 mg/ml) followed by addition of concentrated phosphoric acid. Neutralize with concentrated ammonia to precipitate calcium phosphate. Keep it overnight. Decant and centrifuge. Discard the supernate and washings. Dissolve the precipitate in 8 N nitric acid and pass through a strong anion exchange column preconditioned with 8 N nitric acid at a rate of about 0·5 ml/min. Pass the column with 20 ml 8 N nitric acid. Thorium will be retained while uranium and radium will pass through. Collect the effluent (F_2). Elute thorium with 20 ml of 8 N hydrochloric acid at a rate of about 0·5 ml/min. Analyse the eluate for thorium. Evaporate the effluent F_2. Dissolve the residue in 8 N hydrochloric acid and pass through an anion exchange column preconditioned with 8 N hydrochloric acid at a rate of about 0·5 ml/min. Collect this effluent (F_3). Wash the column with about 20 ml 8 N hydrochloric acid at the rate of about 0·5 ml/min. Radium will pass through the column and uranium will be retained. Combine the washings with the effluent F_3 and preserve for radium analysis. Elute the uranium from the column by 20 ml of 0·1 N nitric acid at a rate of about 0·5 ml/min. Evaporate the elute to dryness and preserve for uranium analysis.

4.5.5 *Radium* — 226 + 224 and 228

4.5.5.1 *Principle of the Methods* — Radium is coprecipitated along with barium *alpha* sulphate and purified. Ra 226 + 224 is counted for α activity using silver activated zinc sulphide α-counter, while Ra 228 being a β-emitter is allowed to decay into actinium-228 till a secular equilibrium is reached. Activity due to actinium-228 is then counted using a G.M. counter.

NOTE — In an undisturbed sample (no radionuclide isolated from the system) an equilibrium between the parent and daughter is established in which the rate of formation of the daughter atom is just equal to the rate of decay of the parent atom. This state is known as secular equilibrium which is attained when the parent has a very long half life compared to the daughter. Mathematically secular equilibrium may be defined as $N_1X_1 = M_2X_2$ where N_1, X_1 and N_2, X_2 are the number of atoms and decay constants of the parent and a daughter respectively. The half life of ^{228}Ra is 6·70 years, while its daughter actinium-228 has a half life of 6·13 hours. Hence a secular equilibrium between the two is reached after about six half lives of actinium 228 that is, after about 36 hours.

4.5.5.2 *Reagents*

For Radium 226 + 224

a) *Nitric acid* — concentrated;

b) *Barium carrier* — 5 mg/ml;

c) *Lead carrier* — 100 mg/ml;

d) *Sulphuric acid* — (1 : 1);

e) *Ethylene diamine tetra-acetic acid* (*EDTA*) — 10 percent. About 10 ml of liquorammonia is added to 10 percent EDTA solution before making up the volume. A clear smell of ammonia should persist; and

f) *Glacial acetic acid.*

For radium — 228

a) *Nitric acid* — concentrated;

b) *Barium carrier* — 5 mg/ml;

c) *Sulphuric acid* — 1 : 1;

d) *Ethylene diamine tetra acetic acid* — 10 percent;

e) *Glacial acetic acid*;

f) *Perchloric acid* — concentrated;

g) *Bismuth carrier* — 10 mg/ml;

h) *Hydrofluoric acid* — concentrated;

j) *Hydrochloric acid* — concentrated; and

k) *Wash solutions*:

1) 14·8 ml concentrated hydrofluoric acid + 34·0 ml concentrated hydrochloric acid diluted to 400 ml with distilled water; and

2) 14·8 ml concentrated hydrofluoric acid + 25·6 ml concentrated nitric acid diluted to 400 ml with distilled water.

4.5.5.3 *Procedure*

a) *General* — Take the solution preserved earlier for radium analysis in **4.5.4.3**, or in case only radium analysis is required in the given water sample, take 4 litres of the water sample filtered through Whatman filter paper 40. Evaporate to dryness. Dissolve the residue in 5 to 10 ml concentrated nitric acid and again evaporate for dryness. Once again dissolve in 5 to 10 ml concentrated nitric acid and evaporate to dryness so that the entire radium present in the sample is converted into nitrate form. Dissolve the residue in water and add 0·5 ml barium carrier (5 mg/ml) and 2 ml lead carrier (100 mg/ml). Precipitate sulphates with 1 : 1 sulphuric acid. Centrifuge and discard the supernate. Dissolve the precipitate in 10 percent ammoniacal EDTA solution, while keeping on a water bath. Add glacial acetic acid and precipitate barium sulphate. Centrifuge and discard the supernate containing lead. Once again dissolve precipitate in 10 percent ammoniacal EDTA solution, and glacial acetic acid and precipitate barium sulphate. Centrifuge and discard supernate. This will remove any traces of lead.

b) *For Radium (226 + 224)* — Take background α counts of a washed, dried and pre-weighed stainless steel disc (about 2·5 to 5·0 cm in diameter). Transfer the precipitate from (a) on to the disc with the help of minimum quantity of distilled water. Dry under an infra red lamp. Fire on a burner flame and cool. Count α activity due to 226 Ra + 224 Ra. Count the reference standard for converting counter per second into disintegrations per second.

c) *For Radium*-228 — To the precipitate of (Ra — Ba) SO_4 in the centrifuge tube obtained in (a) add 1 ml lanthanum carrier (5 mg/ml), 1 ml lead carrier (100 mg/ml) and 0·5 ml bismuth carrier (10 mg/ml). Dry under an infra red lamp. Keep for 36 hours at room temperature for attaining secular equilibrium between radium and actinium. Dissolve the residue in concentrated perchloric acid by heating the centrifuge tube slowly and cautiously. Add acid (1 : 1) to precipitate (Ba, Ra, Pb) SO_4. Centrifuge and transfer the supernate (S_1) to the polyethylene centrifuge tube. Note the time t_1 of the separation of the supernate and precipitate. Dissolve the precipitate in ammoniacal EDTA and reprecipitate (Ba Ra) SO_4 with glacial acetic acid. Centrifuge and discard supernate. Dissolve the precipitate again in ammoniacal ETDA and reprecipitate (Ba Ra) SO_4 with glacial acetic acid, centrifuge and discard supernate and wash the precipitate with distilled water. Transfer the precipitate with water on washed and pre-weighed stainless steel disc. Dry under infra red lamp and fire over flame and weigh to get Ba recovery, say R percent. To supernate S_1 in polyethylene centrifuge tube, add 1-2 ml concentrated hydrochloric acid and 2-3 ml concentrated hydrofluoric acid to get LaF_3 precipitate (228 Ac is carried with it). Centrifuge and discard the supernate. Wash the LaF_3 precipitate once with wash solution (1) and once with (2) to remove traces of bismuth present and finally 2 to 3 times with water. Transfer LaF_3 precipitate on washed preweighed aluminium planchet. Dry under infra-red lamp and weigh the planchet again to get La recovery, say y percent. Count beta activity due to 228 Ac using a G.M. counter. Note the time t_2 at the middle of the LaF_3 counting. Take background and reference standard counts also.

4.5.5.4 *Calculations*

a) *For Radium 226 + 224*

$$\begin{array}{l}\alpha\text{-activity due to}\\ \text{Ra} - 224 + 226\\ \text{Bq/l}\end{array} = \left[\left(\frac{C_s}{t_s} - \frac{C_b}{t_b}\right) \pm \sqrt{\frac{C_s}{t_s^2} + \frac{C_b}{t_b^2}}\right]$$

$$\times \frac{S}{\left(\dfrac{C_{\mathrm{ref}}}{t_{\mathrm{r}}} - \dfrac{C_{\mathrm{b}}}{t_{\mathrm{b}}}\right)} \times \frac{1\,000}{y} \times \frac{1}{V}$$

where the various terms have the same meanings as in **4.5.1.4.**

$$y = \frac{\text{Mass of } \mathrm{BaSO}_4 \text{ ppt in mg} \times \dfrac{137{\cdot}34}{261{\cdot}38}}{\text{Mass of barium carrier in mg}} \times 100$$

b) *For Radium 228*

$$\begin{matrix}\text{Ra — 228} \\ (\text{Bq/l})\end{matrix} = \left[\left(\frac{C_{\mathrm{s}}}{t_{\mathrm{s}}} - \frac{C_{\mathrm{b}}}{t_{\mathrm{b}}}\right) \pm \sqrt{\frac{C_{\mathrm{s}}}{t_{\mathrm{s}}^2} + \frac{C_{\mathrm{b}}}{t_{\mathrm{b}}^2}}\,\right]$$

$$\times \left\{\frac{S}{\left(\dfrac{C_{\mathrm{ref}}}{t_{\mathrm{r}}} - \dfrac{C_{\mathrm{b}}}{t_{\mathrm{b}}}\right)} \times \frac{100}{y} \times \frac{1\,000}{R} \times \frac{1}{V} \times e^{\frac{0{\cdot}693\,(t_2 - t_1)}{6{\cdot}13}}\right\}$$

where the various terms have the same meaning as given in **4.5.1.4.**

$t_2 - t_1$ = time between separation of precipitate and middle of counting in hours

$$R = \text{percent recovery of Ba} = \frac{\text{Mass of } \mathrm{BaSO}_4 \times \dfrac{137{\cdot}34}{261{\cdot}38}}{\text{Mass of Ba carrier add in mg}}$$

$$Y = \frac{\text{Mass of } \mathrm{LaF}_3 \text{ in mg} \times \dfrac{138{\cdot}91}{195{\cdot}90}}{\text{Mass of La carrier added in mg}} \times 100$$

4.5.6 *Thorium (natural)*

4.5.6.1 *Spectrophotometric method*

Principle — Thorium is complexed with thoronal and the optical density of the complex is measured at 545 nm.

Reagents

a) *Perchloric acid* — concentrated,

b) *Thoronal solution* — 1 percent, and

c) *Thorium standard solution* — 1 to 5 μg/ml.

NOTE — Thoronal is 1 — (ortho arseono phenylazo)-2 naphthol — 3, 6 — disulphonic acid

or

2 — (2-hydroxy — 3, 6 disulpho-1 naphthylazo benzene arsonic acid.

Method of preparation of thorium standard solution — Dissolve 3·0 g thorium nitrate [$Th(NO_3)_4$,]·12 H_2O, in 1 litre of distilled water containing 1-2 ml concentrated nitric acid. The strength of this solution is approximately 100 mg Th/litre. To standardize this stock solution take 100 ml solution in a beaker and evaporate to dryness. Dissolve the residue in 100 ml 3·5 percent hydrochloric acid solution. Boil and add slowly 10 percent oxalic acid solution to precipitate thorium oxalate. Add a few ml of oxalic acid in excess for ensuring complete precipitation of thorium oxalate. Cool and keep overnight. Filter through an ashless filter paper (Whatman No. 540). Ignite the precipitate in a furnace between 700 to 800°C as thorium oxide till constant mass of ThO_2 is obtained. Determine the amount of thorium and mark the stock solution in terms of mg Th/ml. Dilute the stock solution as required.

Procedure — Concentrate to a very small volume the eluate obtained in **4.5.4.3**, for determination of thorium, and evaporate to dryness with concentrated perchloric acid 5 to 10 ml, or in case only thorium is to be determined in the water sample, take 4 litres of filtered sample and evaporate it to near dryness. Add 5 to 10 ml concentrated perchloric acid and evaporate to dryness. Evaporate to dryness a known volume of the thorium standard solution (strength 1 to 5 µg/ml) with 5 to 10 ml concentrated perchloric acid (same volume as for the sample). To prepare the reagent blank (without the sample or standard), take about 50 ml of distilled water and evaporate to dryness with 5 to 10 ml of concentrated perchloric acid. To each of the residues from steps above add 1 ml thoronal solution and then add 4 ml perchloric acid (*p*H 1·0). Swirl to dissolve the residues. Measure the optical densities of the sample and standard with respect to reagent blank, using a (wavelength of 545 nm) spectrophotometer.

Calculations — Calculate thorium (natural) concentration in µg/ml using the following equation:

$$\text{Thorium (natural) } \mu\text{g/ml} = \frac{d_1}{d_2} \times S$$

where

d_1 = optical density of the sample with respect to reagent blank,

d_2 = optical density of the standard with respect to reagent blank, and

S = strength of the standard solution µg/ml.

4.5.6.2 *Counting method*

Principle — Thorium is separated from other α-emitting radio nuclides, plancheted and counted using a α counter.

Reagents — same as given **4.5.4.2.**

Procedure — Evaporate to dryness the eluate in **4.5.4.3** (for determination of thorium).

Dissolve the residue in minimum quantity of distilled water and planchet on a stainless steel disc (2·5-5 cm diameter). Dry under an infra red lamp, fire on a burner flame and cool it to room temperature. Count α-activity due to thorium (nat) using an α-counter. Take background and reference standard counts also.

Calculations — Calculate thorium (natural) activity using the equation:

$$\text{Thorium (natural) activity in Bq/l} = \left[\left(\frac{C_s}{t_s} - \frac{C_b}{t_b}\right) \pm \sqrt{\frac{C_s}{t_s{}^2} + \frac{C_b}{t_b{}^2}}\right] \times \left[\frac{S}{\left(\frac{C_{ref}}{t_r} - \frac{C_b}{t_b}\right)} \times \frac{1}{V}\right]$$

where the various terms have similar meanings as given in **4.5.1.4.**

4.5.7 *Uranium (Natural)*

4.5.7.1 *Principle* — Uranium, after concentration by precipitation and separation from other radionuclides using ion exchange method, is converted to its fluoride and fluorescence of the solid fluoride is measured using a fluorometer.

4.5.7.2 *Reagents* — In addition to the reagents already described earlier in section **4.5.4.2** for concentration and separation steps, the following reagents are required for the determination step:

a) *Nitric acid* — 1 N,

b) *Flux sodium fluoride* — Sodim carbonate($NaF : Na_2CO_3 = 15 : 85$), and

c) *Standard uranium solution* — 5mg/ml to 1 μ g/ml.

4.5.7.3 *Procedure for Preparation of standard uranium solution* — Dissolve 1·15 gram pure uranium dioxide (UO_2) in minimum amount of concentrated nitric acid and make up to 1 litre. The strength of this solution will be approximately 1 mg U/ml. Alternately dissolve 2·11 gram of $UO_2(NO_3)_2 . 6H_2O$ in 1 litre of water containing 2 to 3 ml of concentrated nitric acid. To get a stock soultion of about 1 mg U/ml concentration standardize the solution prepared above. To standardize, take about 100 ml stock solution and add excess dilute ammonium hydroxide solution to get ammonium diuranate ($(NH_4)_2U_2O_7$ precipitate. Ignite the precipitate in a furnace between 750 to 800°C till constant mass of UO_3 is obtained. Determine the amount of uranium and mark the stock solution as mg U/ml. Dilute the stock solution as required.

4.5.7.4 *Procedure* — Take the eluate in **4.5.4.3** (for determination of uranium) and evaporate it to a dryness or take about 4 litres of filtered water sample. Concentrate by coprecipitating uranium along with calcium phosphate. Dissolve the precipitate in 8 N nitric acid and pass through an anion exchange column (for example, dowex 1·150 to 300 μm) preconditioned with 8 N nitric acid to adsorb thorium on the column. Evaporate the effluent to dryness and take the residue in 8 N hydrochloric acid. Pass the solution through another similar column preconditioned with 8 N hydrochloric acid to remove radium as effluent. Wash the column and finally elute uranium by about 20 to 30 ml 0·1 N nitric acid. Evaporate the eluate to dryness. Dissolve the residue in 5 ml of 1 N nitric acid. Take seven clean and dry platinum planchets (about 0·5 ml capacity). Using a glass micropipette, pour 0·1 ml 100 λ of the solution above into four planchets (Planchet No. 1, 2, 3 and 4). Add washings of micropipette each time (20 to 30 μl). Add (100 λ) 0·1 ml standard solution with a micropipette into the four planchets (Planchet No. 3, 4, 5 and 6). Leave the planchet No. 7 blank. Dry all, the six planchets with solutions under an infra red lamp. Fire the planchets now on a burner flame. Dry and thoroughly mix the flux. Add one flux pellet (pellet size; diameter 9 mm and thickness 3 mm) into each of the seven planchets using a pelletizer. Fuse the uranium residue along with the flux over the burner flame. Cool the planchet to room temperature. Measure the fluorescence intensity of all the seven planchets (sample, sample + standard; standard; blank).

4.5.7.5 *Calculation* — Calculate the concentration of uranium (natural) in the water sample using the following equations:

a) *Non quenched samples* — Samples containing comparatively low total dissolved solids, say from unpolluted lakes, wells, rivers, etc.

$$\text{Uranium (natural) concentration in } \mu\text{g/l} = \left(\frac{x-b}{z-b}\right) \times S \times \frac{5}{V}; \text{ and}$$

b) *Quenched samples* — Samples of effluents, waste waters and polluted waters containing high total dissolved solids.

$$\text{Uranium (natural) concentration in } \mu\text{g/l} = \left(\frac{x-b}{y-x}\right) \times S \times \frac{5}{V}$$

where

b = fluorescence intensity for flux,

x = fluorescence intensity for sample + flux (average of 1 and 2),

y = fluorescence intensity for sample + standard + flux (average of 3 and 4),

z = fluorescence intensity for standard + flux [$(z - b) = (y - x)$] (average of 5 and 6),

V = volume of sample taken in litres,

S = strength of standard (μg/ml), and

5 = factor coming from the final volume of uranium sample made after dissolving the residue (in 5 ml of 1 N nitric acid).

4.5.8 *Plutonium*

4.5.8.1 *Principle* — Plutonium is first concentrated by precipitation and separated from other radionuclides using ion exchange method. The separated plutonium is then electroplated on a stainless steel disc at *p*H 2·5. It is then counted using a surface barrier detector α-spectrometer.

4.5.8.2 *Reagents* — In addition to the reagents given in **4.5.4.2** for concentration and separation steps, the following reagents are required for the electroplating step :

a) *Sulphuric acid* — 1 N and 0·2 N,

b) *Ammonium sulphate* [$(NH_4)_2SO_4$] *solution* — 4 N, and

c) *Ammonia* (NH_4OH) *solution* — (1 : 1).

4.5.8.3 *Procedure* — Take the residue, preserved in **4.5.4.3** for determination of plutonium, and dissolve it in 0·5 ml of 0·2 N sulphuric acid. Add 3 to 4 ml of 4 N ammonium sulphate solution adjusted to *p*H 2·5 using 1 N sulphuric acid and transfer the plutonium solution into the electro deposition cell (described in **4.2**). Rinse the beaker twice with about 3 ml 4 N ammonium sulphate solution (*p*H 2·5), and add the washings also to the electro deposition cell. Pass the current (0·3 amp at 5 V) through the cell for 2 hours with platinum wire as anode and the stainless steel disc serving as cathode. Add 1 ml of 1 : 1 ammonium hydroxide about one minute before stopping the current to avoid redissolution of electrodeposited Pu into solution. Stop the current and pour the cell solution into another beaker.

Rinse the cell with distilled water and remove the stainless steel disc. Rinse the disc again with distilled water followed by an ethyl alcohol wash. Dry the disc under an infra-red lamp and fire over a burner flame. Count α-activity due to various isotopes of Pu using a surface barrier detector α-spectrometer.

4.5.8.4 *Calculation* — Identify the different isotopes of plutonium (236 Pu, 238 Pu, 239 + 240 Pu and 242 Pu) on the basis of the α energies in the α-spectrum obtained and calculated from the radio chemical recovery of the tracer added (242 Pu or 236 Pu) since the chemical recovery of all the isotopes is expected to be identical. Calculate the α-activity due to plutonium using the following equation:

$$\text{Plutonium (Bq/l)} = \left[\left(\frac{C_s}{t_s} - \frac{C_b}{t_b}\right) \pm \sqrt{\frac{C_s}{t_s^2} + \frac{C_b}{t_b^2}}\right]$$

$$\times \left[\frac{S}{\left(\frac{C_{ref}}{t_r} - \frac{C_b}{t_b} \right)} \times \frac{100}{Z} \times \frac{1}{V} \right]$$

where the various terms have similar meanings as given in **4.5.1.4**, and

Z = percent radio chemical or tracer recovery

$$= \frac{\text{dps (tracer) obtained during counting}}{\text{dps (tracer) added}} \times 100$$

4.5.9 *Americium*

4.5.9.1 *Principle* — Americium is first concentrated by precipitation and separated from other radionuclides using ion exchange method. The separated americium is then electroplated on a stainless steel disc at *p*H 2·5. It is then counted using a surface barrier detector α-spectrometer.

4.5.9.2 *Reagents* — In addition to the reagent already given in **4.5.4.2** for concentration and separation steps, the following reagents are required for the electroplating step :

a) *Sulphuric acid* — (H_2SO_4) 1 N and 0·2N.

b) *Ammonium sulphate* [(NH_4)$_2SO_4$] solution — 4N.

c) *Ammonia* (NH_4 OH) *solution.*

4.5.9.3 *Procedure* — Take the residue preserved in **4.5.4.3** for determination of americium and dissolve it in 0·5 ml of 0·2 N sulphuric acid. Add 3 to 4 ml of 4 N ammonium sulphate solution adjusted to *p*H 2·5 using 1 N sulphuric acid and transfer the americium solution into the electrodeposition/plating cell (described in **4.2**). Rinse the beaker twice with about 3 ml 4 N ammonium sulphate solution (*p*H 2·5) and add the washings also to the electrodeposition cell. Pass the current (0·3 amp at 5 V) through the cell for 2 hours with platinum wire as anode and the stainless steel disc serving as cathode. Add 1 ml of 1 : 1 NH_4OH about one minute before stopping the current to avoid redissolution of electrodeposited americium into solution. Stop the current and pour the cell solution into another beaker. Rinse the cell with distilled water and remove the stainless steel disc. Rinse the disc again with distilled water followed by an ethyl alcohol wash. Dry the disc under an infra-red lamp and fire over a burner flame. Count the α-activity due to 241 Am and 243 Am using a surface barrier detector α-counter.

4.5.9.4 *Calculation* — Identify 241 Am and 243 Am on the basis of α-energies in the α-spectrum obtained, and calculate from the radiochemical recovery of the tracer added 243 Am, since the chemical recovery of both the isotopes is expected to be identical. Calculate α-activity due to americium using the following equation :

$$\text{Americium (Bq/l)} = \left[\left(\frac{C_s}{t_s} - \frac{C_b}{t_b}\right) \pm \sqrt{\frac{C_s}{t_a{}^2} + \frac{C_b}{t_b{}^2}}\right] \times \left[\frac{S}{\left(\frac{C_{ref}}{t_r} - \frac{C_b}{t_b}\right)} \times \frac{100}{Z} \times \frac{1}{V}\right]$$

where the various terms have similar meaning as given in **4.5.1.4.**

$$Z = \text{percent radiochemical or tracer recovery}$$

$$= \frac{\text{dps (tracer) obtained during counting}}{\text{dps (tracer) added}} \times 100$$

4.5.10 *Polonium-210*

4.5.10.1 *Principle* — Polonium is directly deposited on a nickel or a silver disc from acidified water sample. This separation method is very specific and can be carried out in the presence of many other radionuclides. The separated polonium on the disc is then counted using a surface barrier detector α-spectrometer.

4.5.10.2 *Reagents*

a) *Hydrochloric acid* — concentrated,

b) *Nitric acid* — concentrated,

c) *Ascorbic acid* — solid, and

d) *Polonium-208 tracer* (*strength about 1 to 10 dpm*).

4.5.10.3 *Procedure* — Take 4 litres of filtered water sample and add about 200 ml concentrated hydrochloric acid. Evaporate to about 20 ml. Transfer the concentrated solution into a 250 ml beaker along with the distilled water washings. Make up to the final solution volume of about 100 ml. Add 1 to 2 ml of polonium 208 tracer of strength about 10 dpm. Add about 100 mg ascorbic acid. Place the beaker on a water bath and heat to 55°C to 60°C. Rinse the 2·5 cm diameter Ni or Ag disc in concentrated nitric acid followed by rinsing in concentrated hydrochloric acid and a final wash with distilled water. Place the disc in the beaker containing the concentrated sample. Stir the solution while on water bath (55°C to 60°C) vigorously with a mechanical stirrer for about 3 hours the disc should spin and not lie at the bottom of the beaker. Stop stirring, remove the disc, and rinse it with distilled water. Dry under an infra red lamp. Count α-activity due to 210 P_0 and 208 P_0 on both the sides of the disc using surface barrier detector α-spectrometer.

4.5.10.4 *Calculations* — Identify 210 P_0 and 208 P_0 on the basis of their α-energies in the α-spectrum obtained and calculate from the radiochemical recovery of the tracer added.

Calculate the activity due to 210 P_o using the following equation:

$$\text{Polonium (Bq/l)} = \left[\left(\frac{C_s}{t_s} - \frac{C_b}{t_b}\right) \pm \sqrt{\frac{C_s}{{t_s}^2} + \frac{C_h}{{t_b}^2}}\right] \times \left\{\frac{S}{\left(\frac{C_{ref}}{t_r} - \frac{C_b}{t_b}\right)} \times \frac{100}{Z} \times \frac{1}{V}\right\}$$

where the various terms have similar meanings, as given in **4.5.1.4** and

$$Z = \text{percentage radiochemical or tracer recovery}$$

$$= \frac{\text{dps (tracer) obtained during counting}}{\text{dps (tracer) added}} \times 100$$

4.5.11 *Ruthenium (Ru)*

4.5.11.1 *Principle* — Ruthenium is distilled from the water samples as volatile Ruthenium tetra oxide. It is then converted to ruthanium hydrous oxide for purification and then converted to chloride by dissolving in concentrated hydrochloric acid. The purified chloride is then reduced to ruthenium metal by magnesium and β activity of 103 Ru and 106 Ru is counted using a G.M. counter.

4.5.11.2 *Reagents*

a) *Sulphuric acid* (1 : 1),
b) *Ru carrier* — 10 mg/ml,
c) *Potassium permanganate* — solid,
d) *Sodium hydroxide solution* — 6 N,
e) *Ethyl alcohol* — distilled,
f) *Hydrochloric acid* — concentrated, and
g) *Magnesium* — powder.

4.5.11.3 *Procedure* — Take 4 litres of the filtered water sample. Add about 25 ml of sulphuric acid and 2 ml of ruthenium carrier (10 mg/ml). Evaporate the solution to about 25 ml. Transfer the solution and washings into a distillation flask. Add about 1 g of potassium permanganate and connect the vapour passing tube. Submerge the vapour outlet of the distillation unit in a centrifuge tube containing about 40 ml of 6 N sodium hydroxide solution, kept in an ice bath. Control the air inlet into the distillation flask at the rate of 3 to 4 bubbles/sec. Heat the flask gently with a burner till the solution boils and the emanating brown RuO^4 vapours get fully condensed into the sodium hydroxide solution in the centrifuge tube (*see* Fig. 8). Cool the flask. Add 1 g potassium permanganate and boil again to distil any remaining ruthenium. Remove the delivery tube from sodium hydroxide solution in the centrifuge tube and discard the contents of the distillation flask. Add about 3 ml ethyl alcohol in the centrifuge tube and heat the contents on a water bath to precipitate hydrous ruthenium oxide. Centrifuge and discard the supernate. Dissolve the precipitate in about 3 ml concentrated hydrochloric acid and dilute to about 20 ml with water. Add magnesium powder in small portions till black ruthenium metal precipitate and the solution becomes colourless. Add concentrated hydrochloric acid dropwise to dissolve any excess magnesium. Boil, cool and centrifuge.

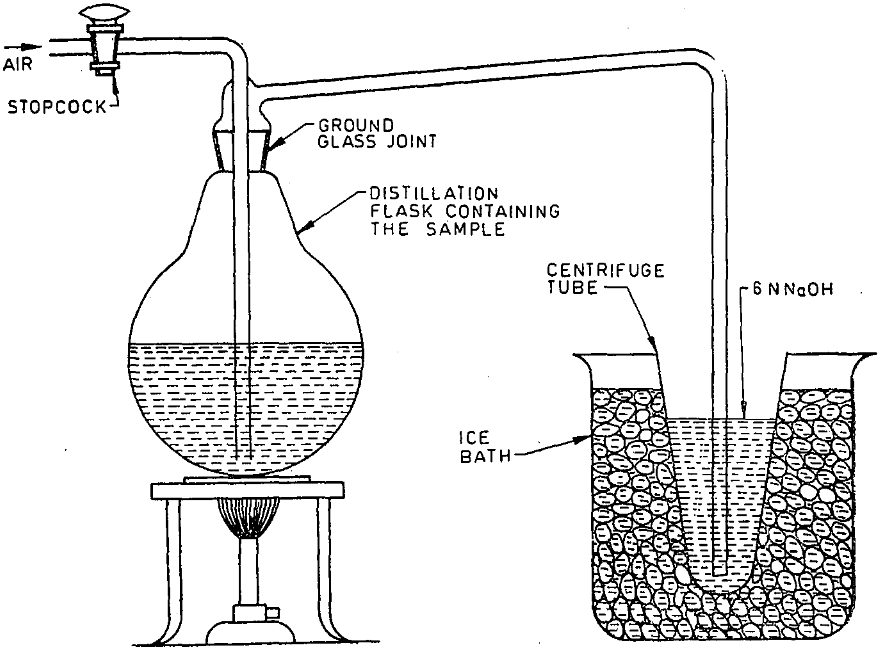

FIG. 8 DISTILLATION ASSEMBLY FOR RUTHENIUM DISTILLATION

Discard the supernate. Wash the precipitate thrice with boiling distilled water and once with ethyl alcohol. Transfer the precipitate with the help of ethyl alcohol on to a washed, dried and pre-weighed aluminium planchet. Dry under an infra red lamp and count β-activity due to 103 Ru and 106 Ru. Keep a 5 mg/cm^2 thickness aluminium absorber over the planchet and count 103 Ru + 106 Rh. Ru 106 being a weak emitter (β energy : 0·030 MeV) will not be counted. 106 Ru can be counted indirectly as 106 Ru (β energy : 3·5 MeV) with which it is in equilibrium. This is carried out by keeping a 250 mg/cm^2 aluminium absorber over the planchet, so that while β-particles due to 103 Ru (energy 0·22 MeV) get completely blocked, only particles due to 106 Ru pass through and get counted. From the observed counts of 106 Ru concentration of 106 Ru can be calculated indirectly.

4.5.11.4 *Calculation*

$$\underset{(\text{ Bq/l })}{\text{Activity due 106 Ru}} = \left[\left(\frac{C_s}{t_s} - \frac{C_b}{t_b}\right) \pm \sqrt{\frac{C_s}{t_s^2} + \frac{C_b}{t_b^2}}\,\right] \times \left[\frac{S}{\left(\frac{C_{ref}}{t_r} - \frac{C_b}{t_b}\right)} \times \frac{100}{y} \times \frac{1}{V}\right]$$

where the various terms have similar meanings as given in **4.5.1.4** and

y = percentage chemical recovery

$$= \frac{\text{mass of Ru metal on the planchet (mg)}}{\text{mass of Ru carrier taken (mg)}} \times 100$$

4.5.12 *Tritium*

4.5.12.1 *Principle* — Tritium is always found to be in equilibrium between water vapour and water. The water sample can therefore be distilled to remove any fluorescence quenching impurities from it. The distillate is then added to the water miscible liquid scintillator and the weak β-activity due to $^3H_1$ (0·018 MeV) is counted by using a liquid scintillation counter.

Alternately tritium may also be first concentrated by employing a prolonged electrolysis procedure using nickel or plantinum electrodes and the concentrate then added to the water miscible liquid scintillator for counting in a liquid scintillation counter. The assembly for electrolysis concentrations is shown in Fig. 9. Electrolysis concentration is generally carried out for drinking water samples where very low tritium levels and comparatively lower amounts of dissolved salts are expected to be present. For effluents and waste water samples this made of concentration is not suitable.

4.5.12.2 *Reagents* — Water miscible liquid scintillator [100 g naphthalene (scintillation grade) + 7 g P.P.O (2,5, diphenyl Oxazole)

0·3 POPOP (2,2 paraphenylene bis-5-phenyl Oxazole, dissolved in 1 000 ml of 1, 4 dioxane) or instagol (a ready made liquid scintillation cocktail).

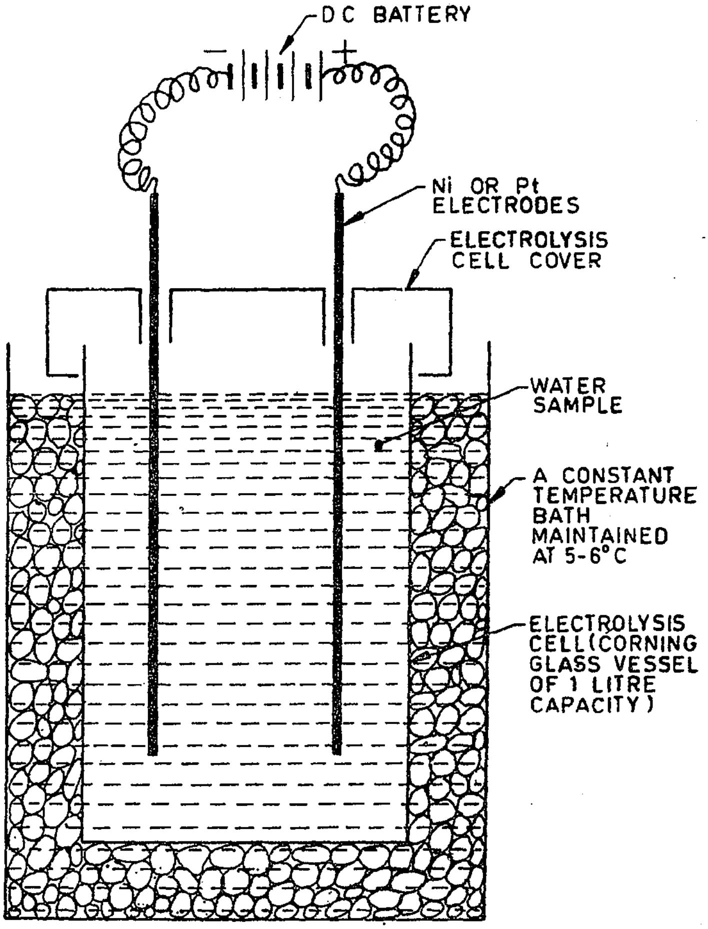
D C BATTERY
−
+
Ni OR Pt ELECTRODES
ELECTROLYSIS CELL COVER
WATER SAMPLE
A CONSTANT TEMPERATURE BATH MAINTAINED AT 5-6°C
ELECTROLYSIS CELL (CORNING GLASS VESSEL OF 1 LITRE CAPACITY)

FIG. 9 ELECTROLYSIS CONCENTRATION ASSEMBLY FOR TRITIUM CONCENTRATION IN WATER SAMPLES

4.5.12.3 *Procedure*

a) *Concentration of evaporation* — Take 5 to 10 ml water sample in porcelain dish (about 9 cm diameter 4 cm depth and 140 ml capacity) and keep it over a hot plate. Invert over the dish a clean and dry glass petri plate. Collect the condensed water from the petri plate in a clean glass vial till its volume becomes 1 to 3 ml. Take a known volume (about 2 ml of the distillate in a clean dried scintillation vial containing 15 ml liquid scintillator. Count β-activity due to $^3H_1$ using a liquid scintillation counter. Take standard (tritiated water) into the scintillation vial and add the liquid scintillator to keep the same water to scintillator ratio. Count the β-activity of the standard. Take the background counts also by taking distilled water in the vial and keeping the same ratio of distilled water to the scintillator.

b) *Concentration by electrolysis* — Take known amount of water sample (about 1 litre) in an electrolysis cell with nickel or platinum electrodes. Make the sample alkaline by adding solid sodium hydroxide so as to get one percent sodium hydroxide solution in the sample. Connect the cell to a D.C. battery and pass a current of 1 amp at 12·0 V. While conducting the electrolysis, the cell should be kept in a cold temperature bath maintained at 5 to 6°C. Reduce the volume of the sample to about 1/4 to 1/5 of the original volume (volume of the concentrate may be about 200 ml). Distil the concentrated alkaline sample completely so as to get condensate completely free of sodium hydroxide (presence of sodium hydroxide causes change in the counting efficiency). Take 2 ml aliquot of the sodium hydroxide free concentrate in a vial containing 15 ml of liquid scintillator. Count the tritium activity present in the aliquot using a liquid scintillation counter.

4.5.12.4 *Calculations*

(a) Tritium, β-activity (Bq/l)

$$= \left[\left(\frac{C_s}{t_s} - \frac{C_b}{t_b}\right) \pm \sqrt{\frac{C_s}{t_s^2} + \frac{C_b}{t_b^2}}\right] \times \left[\frac{S}{\left(\frac{C_{ref}}{t_r} - \frac{C_b}{t_b}\right)} \times \frac{1\,000}{V}\right]$$

where the various terms have similar meanings as given in **4.5.1.4**.

V = volume of distillate taken in the scintillation vial for counting.

b) In case of concentration by electrolysis, calculate original activity using the following equation :

$$C_0 = C\left(\frac{V}{V_0}\right)^{\beta}$$

where

C_0 = original sample activity per ml (to be determined);

C = activity per ml measured in the concentrate;

V_0 = original sample volume in ml taken in the electrolysis cell;

V = volume of the sample in ml after electrolysis; and

β = cell constant, which depends on temperature, electrode distance, voltage and current.

NOTE — Determine the cell constant β by the following process:

Add tritium standard (about 1 000 dpm) into known amount of water (p ml). Count the beta activity of 2 ml aliquot of the above solution by liquid scintillation counter ('x' dpm). Electrolyse the solution after making it alkaline with sodium hydroxide. Continue the electrolysis till about 1/10th of the original volume is obtained. Note the exact volume of the concentrate ('q' ml). Take 2 ml aliquot of the concentrate (after removal of sodium hydroxide by distillation) and count the beta activity ('y' dpm). Calculate the cell constant B by the following formula:

$$\frac{x}{y} = \left(\frac{q}{p}\right)^{\beta}$$

$$\log\left(\frac{x}{y}\right) = \beta \log_{10}\left(\frac{q}{p}\right)$$

$$\text{or } \beta = \log_{10}\left(\frac{x}{y}\right)\Big/\log_{10}\left(\frac{q}{p}\right)$$

The value of β varies from 5 to 8 at low temperatures. In the single stage of electrolysis only about 10 to 15 ml volume reduction can be achieved per day. Hence many stages of electrolysis cell are sometimes required to be employed in series to reduce the time of concentration.

4.5.13 *Cobalt-60*

4.5.13.1 *Principle* — Cobalt in the water sample is separated from most of the other nuclides by precipitating them along with $Zr_3(PO_4)_4$ followed by an anion exchange treatment to remove nickel. Cobalt in the sample is then reduced to the divalent state and precipitated as oxalate. β-activity in the oxalate precipitate is then counted using a G.M. counter.

4.5.13.2 *Reagents*

a) *Anion exchange resin* — 850 µm to 300 µm (Amberite IRA-400, chloride form has been found suitable);

b) *Oxalic acid solution* — saturated;

c) *Hydrochloric acid* — 4 N and 8 N;

d) *Nitric acid* — concentrated;

e) *Sodium sulphite* (Na_2SO_3) — solid;

f) *Cobalt carrier* — 10 mg/ml;

g) *Nickel carrier* — 10 mg/ml;

h) *Zirconium carrier* — 10 mg/ml; and

j) *Disodium hydrogen phosphate.*

4.5.13.3 *Procedure* — Take 4 litre filtered water sample. Concentrate by evaporation to a volume of about 500 ml. Add 1 ml cobalt carrier, 10 ml zirconium carrier and 1 ml nickel carrier. Add 30 ml concentrated nitric acid. Digest over a hot plate to dryness. Dissolve the residue in 5 to 10 ml of 4 N hydrochloric acid. Add the disodium hydrogen phosphate solution (saturated) so as to precipitate zirconium phosphate [$Zr_3(PO_4)_4$]. Centrifuge, wash the precipitate and discard it. Evaporate the supernate along with a distilled water washing to dryness. Dissolve the residue in 5-10 ml of 8 N hydrochloric acid, prepare an anion exchange resin column (column size same as given in **4.5.3.3**) and condition it with 8 N hydrochloric acid for 10 minutes at the rate of 2 to 3 ml per minute, and pass the solution through the column at a rate of about 0·5 ml/min. Nickel will pass through as the effluent (ENi) and cobalt will be retained in the column. Wash the column with about 20 ml of 8 N hydrochloric acid. Add the washings to the effluent. Elute the adsorbed cobalt by using about 30 ml of 4 N hydrochloric acid at a rate of about 0·5 ml/min.

Collect the eluate and evaporate it to a small volume (about 10 ml). Take the above concentrate in a centrifuge tube and add a few milligrams of sodium sulphite (Na_2SO_3) to reduce Co^{3+} to Co^{2+} and precipitate cobalt sulphite. Centrifuge and discard the supernate. Wash the precipitate twice with distilled water and reject washing. Dissolve the cobalt sulphite ($CoSO_3$) precipitate in 4 N hydrochloric acid. Add saturated solution of oxalic acid to precipitate cobalt oxalate. Centrifuge and discard the supernate. Wash the precipitate with distilled water and reject the washings. Transfer the precipitate on to a washed, dried and pre-weighed Whatman No. 42 filter paper (area of the circular filter paper disc is 2·5 cm²). Wash the precipitate with ethyl alcohol and dry at 80 to 100°C in an air oven for about 15 min. Accurately weigh the filter paper disc along with the precipitate and compute the mass of the precipitate. Keep the filter paper disc along with the precipitate on an aluminium planchet and count β-activity due to cobalt-60 by using a G.M. counter. Similarly take the background and reference standard counts also.

4.5.13.4 *Calculations*

$$\text{β-activity due to } {}^{60}\text{Co in Bq/l} = \left[\left(\frac{C_s}{t_s} - \frac{C_b}{t_b} \right) \pm \sqrt{\frac{C_s}{t_b^2} + \frac{C_b}{t_s^2}} \right] \times \frac{S}{\dfrac{C_{ref}}{t_r} - \dfrac{C_b}{t_b}} \times \frac{100}{y} \times \frac{1}{V}$$

where all the terms have similar meanings as given in **4.5.1.4**.

y = percent chemical recovery

$$= \frac{\text{Mass of cobalt precipitate in mg} \times \frac{58{\cdot}94}{146{\cdot}94}}{\text{Mass of cobalt carrier taken in mg}} \times 100$$

4.5.13.5 Cobalt-60 and also cobalt-58 (if required to be determined in water sample) may be simultaneously determined by taking γ spectrum using a sodium iodide (NaI, Ti) crystal detector in conjunction with a r-spectrometer. [Cobalt-60 has two gamma peaks, namely: 1·17 and 1·33 Mev while cobalt-58 has only one gamma peak at 0·61 Mev.]

4.5.14 *Nickel-63*

4.5.14.1 *Principle* — Nickel in the water sample is separated from cobalt using an anion exchange column. It is then purified from cerium, if any, by complexing with ammonia. The purified nickel is precipitated as dimethyl glyoxime complex and after converting to oxalate is counted for β-activity by using a liquid scintillation counter [^{63}Ni β energy ≃ 0·065 Mev].

4.5.14.2 *Reagents* — Reagents required for separation of nickel from cobalt are same as given in **4.5.13.2**. Additional reagents required are given below:

a) *Concentrated ammonia;*

b) *Dimethyl glyoxime solution* — one percent in distilled ethyl alcohol;

c) *Liquid scintillator* — (7·84 g), bis-methylstyryl benzene 0·16 g in 1 : 1 dioxane toluene (1 000 ml); and

d) *Alcohol (distilled ethyl alcohol)* — saturated oxalic acid solution.

4.5.14.3 *Procedure* — Take the effluent ENi from **4.5.13.3**. Add minimum quantity of ammonia solution so as to precipitate cerium present, if any as cerium hydroxide $Ce(OH)_3$. Centrifuge, wash the precipitate with distilled water, and then discard it. Take the supernate along with the washings and add sufficient amount of dimethyl glyoxime solution to quantitatively precipitate nickel dimethyl-glyoxime complex (NiDMG). Filter the NiDMG precipitate through Whatman No. 541 filter paper. Wash the precipitate once with water. Dry under infra-red lamp and burn the filter paper along with precipitate in a silica crucible to destroy NiDMG complex (no carbon is left in the crucible). Dissolve the nickel oxide (NiO) in concentrated hydrochloric acid. Precipitate nickel oxalate (NiC_2O_4) with standard oxalic acid solution. Centrifuge and discard the supernate. Wash the precipitate with alcohol to remove traces of hydrochloric acid. Transfer precipitate from centrifuge tube into counting vial with liquid scintillator using small portions of 15 ml scintillator. Count the *beta*-activity due to Ni-63 using a liquid scintillation counter. Take background and internal reference standard counts also.

NOTE — Conversion of reddish coloured nickel dimethyl glyoxime complex to oxalate is essential so as to avoid colour quenching in β-scintillation counting.

4.5.14.4 *Calculations*

$$\text{Activity due to } {}^{63}\text{Ni in Bq/l} = \left[\left(\frac{C_s}{t_s} - \frac{C_b}{t_b} \right) \pm \sqrt{\frac{C_s}{t_s^2} + \frac{C_b}{t_b^2}} \right] \times \frac{S}{\dfrac{C_{ref}}{tr} - \dfrac{C_b}{t_b}} \times \frac{1}{V}$$

where all the terms have similar meanings as given in **4.5.1.4.** Complete chemical recovery is assumed.

4.5.15 *Iron 59, Zinc 63 and Cerium 141 + 144 (γ emitters)*

4.5.15.1 *Principle* — These nuclides are separated from each other by preferential precipitation and adsorption on an anion exchange column and finally counted by a NaI-Tl detector used in conjunction with a gamma spectrometer.

4.5.15.2 *Reagents*

a) *Sodium hydroxide solution* — 4 M,
b) *Sodium carbonate solution* — saturated,
c) *Iron carrier* — 10 mg/ml,
d) *Zinc carrier* — 10 mg/ml,
e) *Cerium carrier* — 10 mg/ml,
f) *Ammonium phosphate solution* ($(NH_4)_3PO_4$) — saturated,
g) *Nitric acid* — fuming and dilute (8 N),
h) *Ammonium hydroxide* — concentrated,
j) *Hydrochloric acid* — 8 N and 0·1 N,
k) *Strong anion exchange resin* — *See* Note, and
m) *Hydroiodic acid* (HIO_3) — 10 percent solution.

NOTE — Domex — 1 (300 to 150 µm) has been found suitable.

4.5.15.3 *Procedure* — Take 4 litres of filtered water sample. Add 1 ml iron carrier, 1 ml zinc carrier and 1 ml cerium carrier. Add sodium hydroxide solution so as to make the sample alkaline and precipitate carbonate by adding saturated sodium carbonate solution. Keep for 4 to 5 hours to settle the precipitate. Decant and centrifuge the slurry and collect the supernate. Wash the precipitate (P_1) with distilled water and add the washings to the supernate. To the supernate containing zinc add ammonium phosphate solution to precipitate zinc phosphate [$Zn_3(PO_4)_2$]. Keep it overnight. Decant and centrifuge the slurry. Discard the supernate.

Dissolve the [Zn^3 (PO^4 $)_2$] precipitate in about 5 ml of dilute nitric acid (8 N). Take the solution in a plastic vial and count γ due to Zn using a counting set-up. Take the precipitate P_1 containing carbonates of iron and cerium, and dissolve it in 5 to 10 ml of 8 N nitric acid.

Pass the solution through an anion exchanger column preconditioned with 8 N nitric acid for 10 minutes at a rate of about 0·5 ml/min, (plutonium and thorium if present in the water sample will be retained on the column). Wash the column with about 20 ml of 8 N nitric acid and add the washing to the effluent. Cool the effluent in an ice bath and add fuming nitric acid (75 to 80 percent) to remove any barium and strontium, if present in the sample. If any precipitate is observed centrifuge, and discard the precipitate. Evaporate the supernate. Dissolve the residue in 8 N hydrochloric acid. Pass the solution through an anion exchange column preconditioned with 8 N hydrochloric acid. Cerium will pass through while iron will be retained in the column. Wash the column with about 20 ml of 8 N hydrochloric acid and add the washings to the effluent. To the effluent add sufficient amount of concentrated ammonia solution to precipitate cerium hydroxide. [Ce (OH^3)]. Centrifuge, wash the precipitate and discard the supernate along with the washings. Dissolve the Ce (OH $)^3$ precipitate in 5 to 10 ml of concentrated nitric acid.

Add 5 to 10 ml of 10 percent solution of hydroiodic acid to separate cerium as cerium iodate [Ce (IO_3 $)_3$], precipitate from other rare earths. Dissolve the Ce (IO_3 $)_3$ precipitate in 5 to 10 ml 8 N hydrochloric acid at about 70°C. Take the solution in plastic γ counting vial and count activity of 141 Ce + 144 Ce using NaI (Tl) crystal detector in conjunction with a γ-spectrometer. (Ce-141 γ energy 0·145 MeV); (Ce—144 γ energy 0·034 to 0·134 Mev). Elute the adsorbed iron from the column with about 30 ml of 0·1 N hydrochloric acid at the rate of about 0·5 ml/min. To the eluate add sufficient ammount of concentrated ammonia to precipitate ferric hydroxide [Fe (OH $)_3$]. Centrifuge and discard the supernate. Dissolve ihe precipitate in 5 to 10 ml dilute hydrochloric acid. Take the solution in plastic γ-counting vial and count gamma using NaI (Tl) crystal detecter in conjuction with a γ-spectrometer (γ-energy for $^{59}{}_{\mathrm{Fe}}$1·1 and 1·29 Mev). Count the reference standards for all the three nuclides, namely $^{59}{}_{\mathrm{Fe}}$, $^{65}{}_{\mathrm{Zn}}$ and $^{141}{}_{\mathrm{Ce}}$ separately so as to convert the counts per second into disintegrations per second. Take background counts.

4.5.15.4 *Calculations*

Calculate the gamma activity due to $^{59}{}_{\mathrm{Fe}}$, $^{65}{}_{\mathrm{Zn}}$, or $^{141}{}_{\mathrm{Ce}} + {}^{144}{}_{\mathrm{Ce}}$ using the following equations:

$$\begin{array}{l}{}^{59}\mathrm{Fe}/{}^{65}{}_{\mathrm{Zn}}/141 + {}^{144}{}_{\mathrm{Ce}} \\ \quad + {}^{144}{}_{\mathrm{Ce}} \text{ activity in Bq/l}\end{array} = \left[\left(\frac{C_s}{t_s} - \frac{C_b}{t_b} \right) \pm \sqrt{\frac{C_s}{t_s{}^2} + \frac{C_b}{tb^2}} \right]$$

$$\times \left[\frac{S}{\left(\frac{C_{ref}}{t_r} - \frac{C_b}{t_b} \right)} \times \frac{1}{V} \right]$$

where all the terms have similar meaning as given in **4.5.1.4.** Complete chemical recovery is assumed.

NOTE — If ^{141}Ce and ^{144}Ce are separately required to be evaluated, take the gamma spectrum. The individual isotopes are identified on the basis of gamma energy. The area under the peak (total counts) on comparing with reference standards gives the disintegrations per second which on dividing by total volume (litres) taken will give the γ activity in Bq/l.

(*Continued from page* 2)

Panel for Methods of Test for Water and Effluents, CDC 26 : P1

Convener	*Representing*
Dr B. K. Handa	Central Ground Water Board, New Delhi
Members	
Shri Y. P. Kakar (*Alternate* to Dr B. K. Handa)	
Dr J. A. Barve	Bayer (India) Ltd, Bombay
Shri S. K. Bhattacharjya	Ion Exchange (India) Ltd, Bombay
Shri A. M. Kulkarni (*Alternate*)	
Shri B. B. Bose	Geological Survey of India, Calcutta
Shri Yudhistir (*Alternate*)	
Dr Harish Chandra	Industrial Toxicology Research Centre (CSIR), Lucknow
Shri K. M. Kavadia	Excel Industries Ltd, Bombay
Shri A. G. Lakhani	State Public Health Laboratory, Pune
Dr (Shrimati) L. D. Nevaskar (*Alternate*)	
Shri S. D. Makhijani	Central Board for the Prevention & Control of Water Pollution, New Delhi
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Shri M. V. Nanoti (*Alternate*)	
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Shri U. K. Tiwari	Madhya Pradesh Pradushan Niwaran Mandal, Bhopal
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Dr (Shrimati) Z. R. Turel	The Institute of Science, Bombay
Shri K. C. Mahindra (*Alternate*)	

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	1 N = 1 kg.m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2